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Annual Report
Grant No. N00014-85-K0179
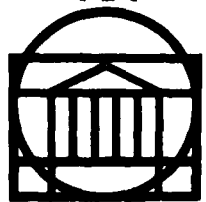September 1, 1987 - September 30, 1988

INVESTIGATION OF MICROSTRUCTURAL FACTORS THAT CAUSE LOW FRACTURE TOUGHNESS IN SILICON CARBIDE WHISKER/Al ALLOY COMPOSITES

Submitted to:

Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217-5000

Attention: Dr. Steven Fishman
Non-Metallic Materials
Code 1131N

Submitted by:

F. E. Wawner
Research Professor

R. D. Schueller
Graduate Research Assistant

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Report No. UVA/525398/MS89/103

October 1988

SCHOOL OF ENGINEERING AND

APPLIED SCIENCE

DEPARTMENT OF MATERIALS SCIENCE

UNIVERSITY OF VIRGINIA

CHARLOTTESVILLE, VIRGINIA 22901

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REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION	1b. RESTRICTIVE MARKINGS
Unclassified	None

2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT
	Approved for public release; distribution
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	unlimited

4. PERFORMING ORGANIZATION REPORT NUMBER(S)

UVA/525398/MS89/103

5. MONITORING ORGANIZATION REPORT NUMBER(S)

6a. NAME OF PERFORMING ORGANIZATION	6b. OFFICE SYMBOL (If applicable)	7a. NAME OF MONITORING ORGANIZATION
University of Virginia Dept. of Materials Science		Office of Naval Research Resident Representative

6c. ADDRESS (City, State, and ZIP Code)	7b. ADDRESS (City, State, and ZIP Code)
Thornton Hall Charlottesville, VA 22901	818 Connecticut Ave., N.W. Eighth Floor Washington, DC 20006

8a. NAME OF FUNDING/SPONSORING ORGANIZATION	8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
Office of Naval Research		N00014-85-K0179

8c. ADDRESS (City, State, and ZIP Code)

800 N. Quincy Street
Arlington, VA 22217-5000

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification)

Investigation of Microstructural Factors that Cause Low Fracture Toughness in Silicon Carbide Whisker/Al Alloy Composites

12. PERSONAL AUTHOR(S)

F. E. Wawner/R. D. Schueller

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Annual	FROM 09/01/87 TO 09/30/88	1988, October 4	

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This program deals with determining factors that influence strength, ductility, fracture toughness, and elongation to failure in SiC whisker/Al alloy composites. Specifically, a microstructural study is being made in an attempt to define extrinsic and intrinsic factors that control fracture and thus influence properties.

One of the more important products produced by ACMC from SiC$_w$/Al composites has been the rolled sheet. The response of the material to secondary processing and thermo-mechanical treatment is of utmost importance with respect to material quality. The present study emphasizes microstructural-mechanical property characterization of the rolled material.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
☑ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT ☐ DTIC USERS	Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL
Dr. Steven Fishman		

DD FORM 1473, 84 MAR

83 APR edition may be used until exhausted.
All other editions are obsolete

It has been confirmed in this study that when Al2124 15% SiCw is hot rolled it loses a good portion of its strength. It has been found that this loss in strength is mainly due to overaging of the precipitation hardenable matrix. Evidence shows that large Al_2Cu precipitates grow as the material is allowed to cool down after the rolling step.

It has also been confirmed that the strength of the material can be regained by subjecting it to a T6 heat treatment (1 hour at 500°C, CWC, and aged at 150°C for 8 hours). Experiments show that the strength is regained mainly because the heat treatment disperses the precipitates. However, it has been discovered that this rather long heat treatment sequence can be made much shorter due to the high dislocation density of this material. The precipitates can be dispersed by solutionizing the material at 500°C for very short periods of time (as short as 1.5 minutes) followed by cold water quenching. Likewise, it has been found that only a very simple aging process is needed to obtain peak strength. Artificial aging at 150°C is not necessary. The material need only be aged at room temperature for a time of 7 hours or more. Thus industry should be able to heat treat the rolled composite material much more efficiently and inexpensively than a T6 heat treatment would permit.

The large precipitates weaken the composite in three basic ways:

1) The growth of large precipitates can weaken the material by removing the small precipitates which are needed to help prevent dislocation motion, thus slip. Therefore, the shear strength of the matrix is reduced which results in a larger critical whisker length. Thus shorter whiskers are unable to carry as much load and the material is weakened.

2) The large precipitates also tend to create large voids in the composite which then act as crack initiation sites and also provide low energy crack paths.

3) Evidence shows that the precipitates tend to nucleate at dislocations near the interface. Therefore, the precipitates tend to grow around the whiskers and envelope them, thus rendering the strengthening effect of the whiskers useless.
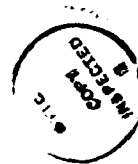
It was shown that the Al2124 matrix provided a much stronger composite than a pure aluminum matrix, at least up to a temperature of 400°C. At temperatures higher than this it appears as if precipitation strengthening offers no contribution to the strength. It has also been shown that the reinforcement itself offers no contribution to the strength at temperatures above 300°C, due to very low shear strengths in the matrix at these high temperatures. Through elevated temperature shear tests it was shown that the main reason for the drop in strength at higher temperatures was due to a rapid decrease in shear strength of the matrix.

It was also found that not only does the T6 heat treatment increase the strength of the rolled composite but it also increases the room temperature ductility. The AR material has very poor ductility because the large precipitates initiate cracks, which lead to tensile fracture, before much plastic deformation can take place. However, the ductility of the AR material increases with temperature to a maximum near 350°C, while the ductility of the T6 material decreases to a minimum at a temperature of 300°C. The precipitates are also thought to be responsible for these effects.

The extruded Al2124 15% SiCw composite was much less homogeneous than the hot rolled material. Precipitates were observed to grow in linear groups along the extruded direction apparently due to variations in plastic deformation.

TABLE OF CONTENTS

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SECTION I
INTRODUCTION

The stigma of high cost which has inhibited the use of metal matrix composite materials for many years is slowly being removed. I... roved manufacturing techniques for the reinforcements and composites, higher volume usage, and a realization that lifetime factors and efficiency improvements must be considered in an overall economic evaluation have led to increased incorporation of the materials into present day and future design concepts.

Additionally, many of the conventional metals and their alloying components (i.e., additives such as Ti, Cr, Co) are primarily imported from potentially unstable nations and have been placed on a critical materials list. For this reason, replacement of these materials may be a forced necessity in the very near future. Metals matrix composites are strongly being considered to fulfill this role.

One such materials system that has moved rapidly from its laboratory inception to commercialization is a silicon carbide whisker reinforced aluminum composite material. This material is composed of typically 15-20 volume percent whiskers in various aluminum alloy matrices and has demonstrated strength values that equal or exceed most Al alloys and a Young's modulus that is 75% higher (18×10^6 psi [124 GPa]). These properties are quite similar to those obtained for many titanium alloys and at a lighter weight, hence, specific properties for the whisker reinforced material are even more impressive. These properties along with additional advantages of being able to use conventional forming methods such as extrusion, forging, rolling, etc.

make the material attractive from an economic as well as a convenience standpoint.

The development of SiC whisker reinforced composite material has followed a logical progression in that baseline mechanical property data has been established, as well as limited microstructural characterization. As with many materials, success comes quickly in the early stages of development only to succumb to more subtle problems later on. These subtleties are overcome only by completely understanding the system. Most frequently the limitations are microstructurally related and on an atomic level. Hence, correlation of specimen history and properties with microstructural observations can lead to an understanding which could extend the range of properties for the material.

This program deals with determining factors that influence strength, ductility, fracture toughness, and elongation to failure in SiC whisker/Al alloy composites. Specifically, a microstructural study is being made in an attempt to define extrinsic and intrinsic factors that control fracture and thus influence properties. The information derived will then be used to make recommendations for upgrading the properties of this important materials system.

Past microstructural studies have indicated that constituent particles in the 3-5 micron size range dominate failure in the present composite systems of interest (i.e. SiC/2124 and SiC/6061). These particles, identified through X-ray analysis as: Al_2CuMg, $Al_{20}Mn_3Cu_2$, and $FeCuMnAl_6$, definitely influence fracture toughness since they generally are brittle and form in critical locations, such as along whisker-matrix interfaces and along grain boundaries. These particles are also found to form in areas where whiskers are

in contact.

Minimizing these detrimental particles is a first step toward improved fracture toughness. Improved extrusion and rolling methods developed at Advanced Composite Materials Corporation have greatly improved whisker distribution throughout the matrix. With better whisker distribution, the probability of precipitate formation at whisker-whisker contact areas is greatly decreased. In addition to minimizing precipitate formation sites, matrix alloy chemistry has been altered to reduce the amount of elements present (i.e. Fe, Mn, Mg, Cu), which are the major components of these constituent particles.

One of the more important products produced by ACMC from SiC_w/Al composites has been the rolled sheet. The response of the material to secondary processing and thermo-mechanical treatment is of utmost importance with respect to material quality. The present study emphasizes microstructural-mechanical property characterization of the rolled material.

SECTION II
RESULTS AND DISCUSSION

EFFECTS OF HOT ROLLING ON AL2124 15% SiCw COMPOSITES

Harris and Wawner (1) noted in an earlier paper that when an Al2124 composite with SiC reinforcement was hot rolled it lost some of its strength. When this composite was subsequently heat treated in the T6 condition its strength was substantially increased. (Note: the T6 condition consists of holding the material at $500^{\circ}C$ for 1 hour, CWC, and aged at $150^{\circ}C$ for 8 hours.) Experiments seemed to show that the strength decrease was a result of interfacial damage which may have occurred during the rolling step. The subsequent heat treatment was noted to heal this interfacial damage, thus restoring the strength.

It has been confirmed in this study that hot rolling does indeed weaken the Al2124-SiC composite, and that a subsequent T6 heat treatment does restore the strength. The following table is the results of room temperature tensile tests performed under different conditions.

Ultimate Tensile Strength
Rolled Al2124-15% SiCw
Room Temperature

Condition	UTS (MPa)	(Ksi)
As Received Transverse	353	51.2
T6 Transverse	557	80.8
As Received Longitudinal	447	64.8
T6 Longitudinal	678	98.3

It is seen that in both the transverse and the longitudinal directions the strength is increased by about 55% after the heat treatment, which is a significant amount. Also, the fact that the strength is increased by almost the same percentage in both the transverse and longitudinal directions seems to suggest that interfacial damage may not be the main cause of the strength loss in this system. Healing of the interfaces would result in more of a one dimensional effect. Thus, one would expect a larger increase in strength in the longitudinal direction. Instead, this data seems to imply a homogeneous effect, which would occur with changes in the matrix.

It has been found in this study that the major cause of weakening due to hot rolling is not from interfacial damage but is instead from overaging of the matrix. The composite material is hot rolled at temperatures in excess of $300^{\circ}C$ and is then allowed to cool in air. It is during this short cooling time that large precipitates are allowed to grow and weaken the composite.

OBSERVING AND IDENTIFYING THE PRECIPITATES

The precipitates can be seen in the AR material by polishing down and ion milling small samples of the material. These surfaces were then studied on the scanning electron microscope (SEM). Figures 1 and 2 are photomicrographs of AR material taken in the backscattering compositional mode (compo). The compo mode shows heavier elements with lighter contrast. The precipitates can be seen rather easily as bright clumps in the matrix, which implies that they consist of heavier elements than aluminum. The precipitates are relatively large with a diameter from 3-5 μm.
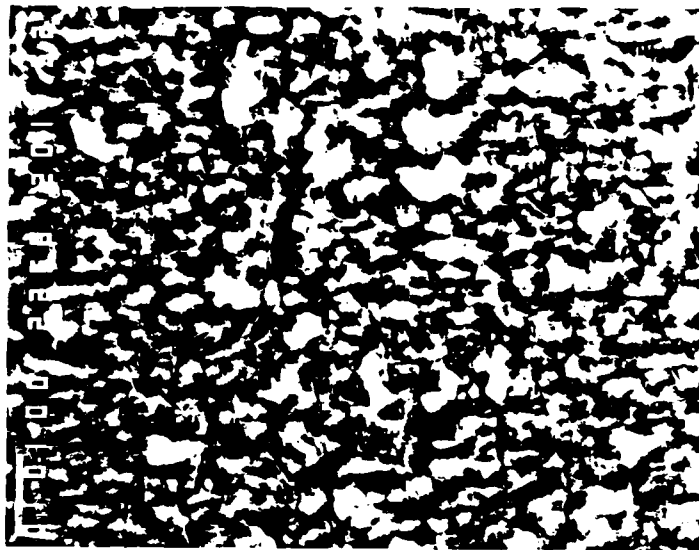
Figure 1.   SEM Backscattered Compositional Mode, 1000x
            As Received material: Large precipitates
            are shown with lighter contrast.

Figure 2.   BEI Compo,   2000x,   AR material: Precipitates
            are about 5 μm in diameter.

Figure 3 and 4 on the other hand, are photomicrographs of T6 material also taken in the compo mode. It is readily noticed that no large precipitates exist. Instead they seem to be dispersed rather evenly throughout the matrix.

Energy dispersive x-ray analysis was performed on the precipitates and it was found that they consisted of copper and aluminum (the x-ray spectrum is shown in fig 5). A semi-quantitative analysis was performed and showed the precipitates to be ⊖ phase Al_2Cu. (see Figure 6)

DISPERSION OF THE PRECIPITATES

Basic calculations were made for the diffusion of copper in aluminum in order to help confirm that these precipitates can be dispersed at a solutionizing temperature of 500°C in one hour's time. The activation energy and the diffusivity constant for the diffusion of Cu in Al in the temperature range 433-652°C were given in the CRC Handbook of Chemistry and Physics. as $Q = 32.27$ kcal/mole and $D_o = 0.647$ cm^2/sec, respectively.(2) Therefore, at the solutionizing temperature of 500°C the diffusion coefficient, D, is

$$D = D_o \exp (Q / RT)$$
$$= 5.57 \times 10^{-10} cm^2/sec$$

The average distance, d, that a copper atom will travel in aluminum can be expressed as

$$d = \sqrt{D * t}$$
$$= 1.42 \times 10^{-3} cm$$
$$= 14 \ \mu m$$

Figure 3.   BEI Compo   1000x
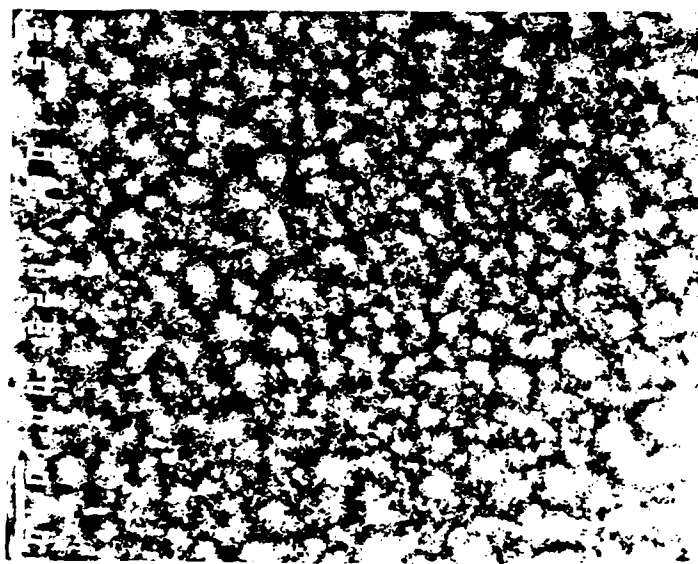T6 material: Precipitates are dispersed.

Figure 4.   BEI Compo   1000x
T6 material: Precipitates are dispersed.

Figure 5. X-ray spectrum of a precipitate.

TN-5500 University of Virginia   THU 16-JUN-88 00:27
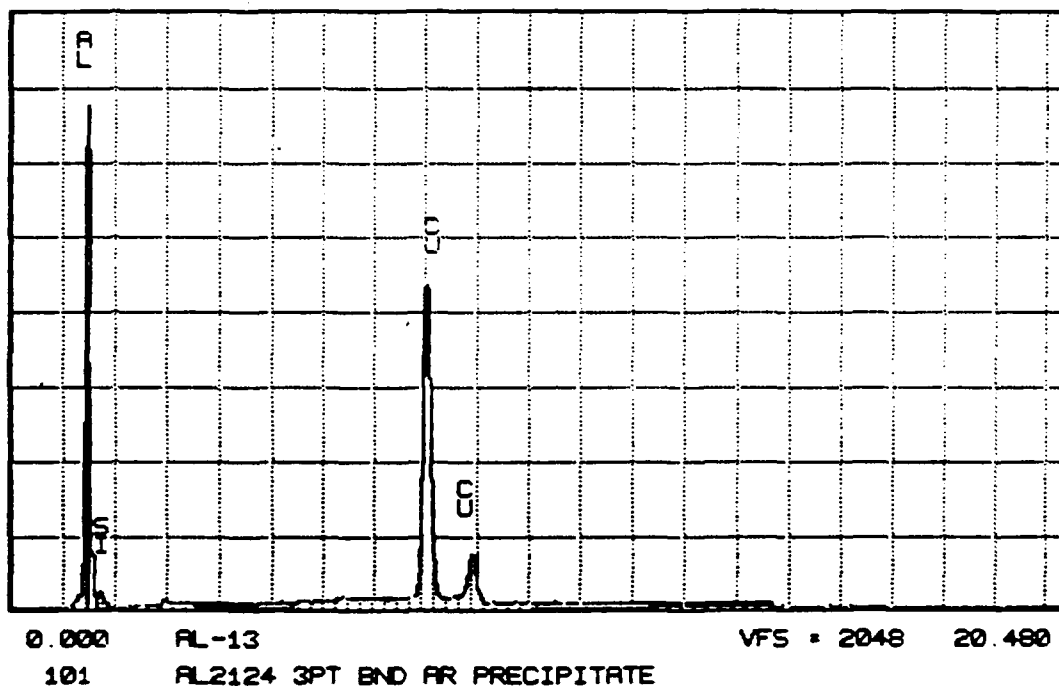Cursor: 0.000keV = 0   ROI   (1)10.170:12.340

0.000   AL-13   VFS = 2048   20.480
101   AL2124 3PT BND AR PRECIPITATE

Figure 6. Semi-quantitative analysis.

SEMI-QUANTITATIVE ANALYSIS: AL2124 3PT BND AR PRECIPITATE
EL   NORM. K-RATIO

AL-K 0.23337 +- 0.00162
MG-K 0.00679 +- 0.00029
CU-K 0.76219 +- 0.00583
SI-K 0.08774 +- 0.00028

ZAF CORRECTION   25.00 KV   47.60 Degs

No. of Iterations 5

| ----	K	[Z]	[A]	[F]	[ZAF]	ATOM.%	WT.%	
AL-K	0.223	0.957	2.831	0.999	2.710	60.74	41.43	*
MG-K	0.006	0.917	4.088	0.991	3.720	2.86	1.73	*
CU-K	0.762	1.057	0.999	1.000	1.057	34.17	55.25	
SI-K	0.007	0.925	3.223	0.999	2.981	2.24	1.58	*

* - High Absorbance

Looking back at Figures 2 and 3, it is seen that 14μm is large with respect to the precipitate size and is about equal to the precipitate spacing. Thus, according to these calculations, the precipitates can, theoretically, be dispersed with the T6 heat treatment. In fact, the diffusion will be much faster in the composite than in the matrix alone due to the high dislocation density of the composite (approximately 10^{13} m^{-2})(3). The dislocation density is much higher in the composite since there is a large difference (10:1) in the thermal expansion coefficients between that of the matrix and the reinforcement. Many more dislocations may also be created due to work hardening which may take place during the rolling step. It is these dislocations which create high speed diffusion paths. Therefore, one would expect the precipitates to be dispersed in less than an hour's time.

In order to get an idea of the speed of diffusion, AR material was solutionized at 500°C for different periods of time. This material was then tensile tested after 24 hours of aging at room temperature. Figure 7 is the graph of strength vs. solutionizing time. It is readily seen that it takes only about 3 minutes to disperse the precipitates and restore the strength of the material! Obviously the high density of dislocations in the rolled Al2124-SiCw composite act to greatly speed up the diffusion in the material.

The high speed of diffusion in the rolled Al2124-SiCw composite system also suggests that the T6 aging condition (8 hours at 150°C) may not be necessary to reach peak strength. Instead, simple room temperature aging was investigated. Samples were solutionized and allowed to room temperature age for different periods of time before being

Figure 7.

UTS vs. SOLUTIONIZING TIME
Rolled AL2124 15% SiCw

Samples were solutionized at 500 degrees celcius, CWC, and then room temperature aged for 24 hours.
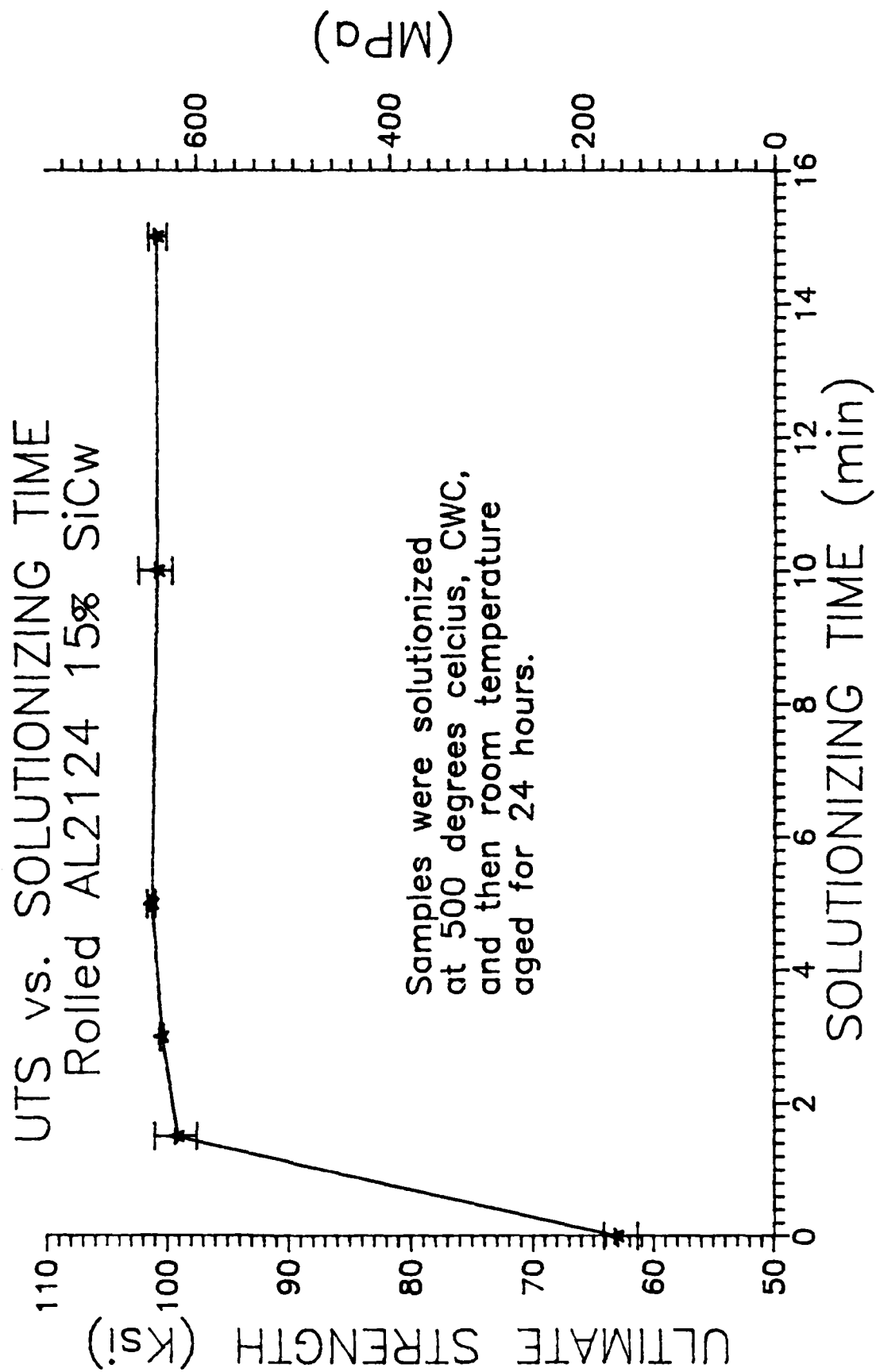
tested. Figure 8 shows the results of this experiment. It can be seen that after only 7 hours of room temperature aging the strength has increased to within 2% of the peak strength. Thus the artificial aging at 150°C is not necessary for this composite system.

GROWTH OF PRECIPITATES UPON COOLING

In order to show that the precipitates grow as the material cools down after hot rolling the following experiment was performed: The AR material was solutionized at 500°C for two hours to ensure full dispersion of the precipitates. The samples were then allowed to cool rather steadily from 500°C to 150°C for different periods of time. At least three tensile tests were done for each condition.

Figure 9 is a plot of ultimate tensile strength vs. cooling time. The error bars show the range of strengths received and when none are shown the scatter is smaller than the point marker. It is apparent that the strength of the material drops off rather steeply with cooling time and then seems to converge to a minimum strength. At a cooling time of just 11 minutes the strength has dropped from 687 MPa (99.7 ksi) down to 456 MPa (66.2 ksi). If the material is allowed to cool for 1 hour the strength drops to 445 MPa (64.5 ksi) which is essentially the same as the AR strength of 441 MPa (63.9 ksi).

This observation alone suggests that interfacial damage is not a significant factor in the decline of strength after hot rolling in the Al2124-SiC system. Any interfacial damage would have healed during the solutionizing step, and the following cooling time would not have an adverse effect on the strength. Therefore, the strength would not drop all the way down to the AR strength upon slow cooling if interfacial damage and healing were involved.
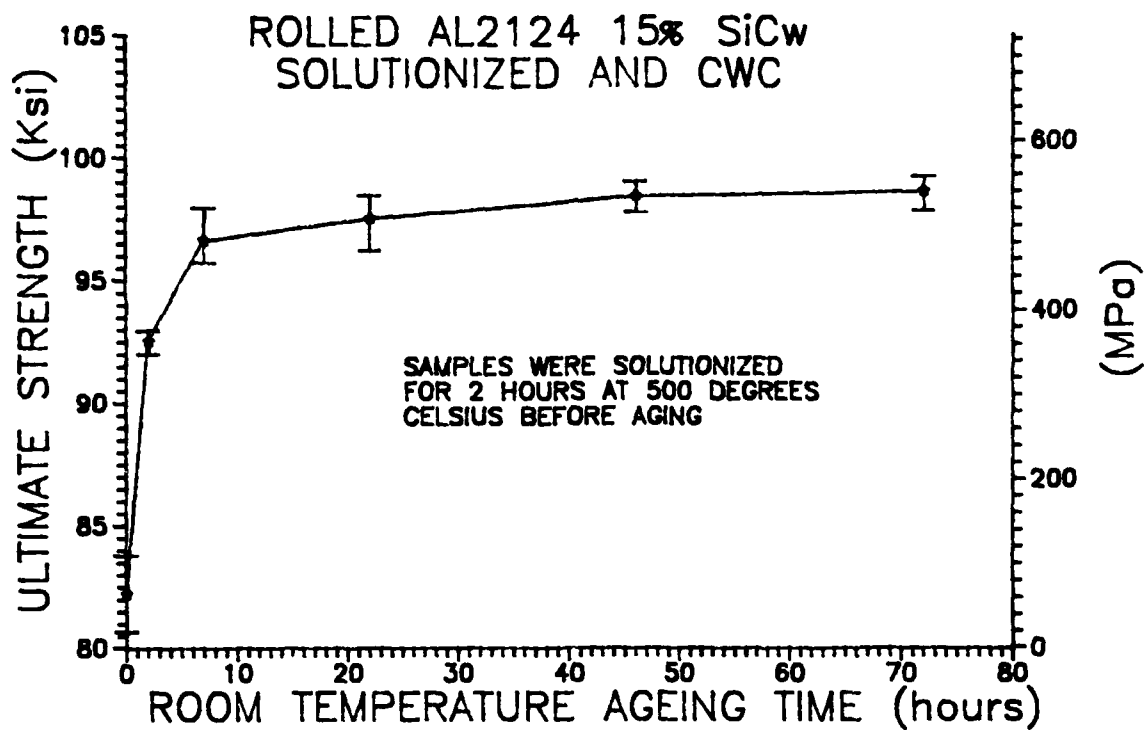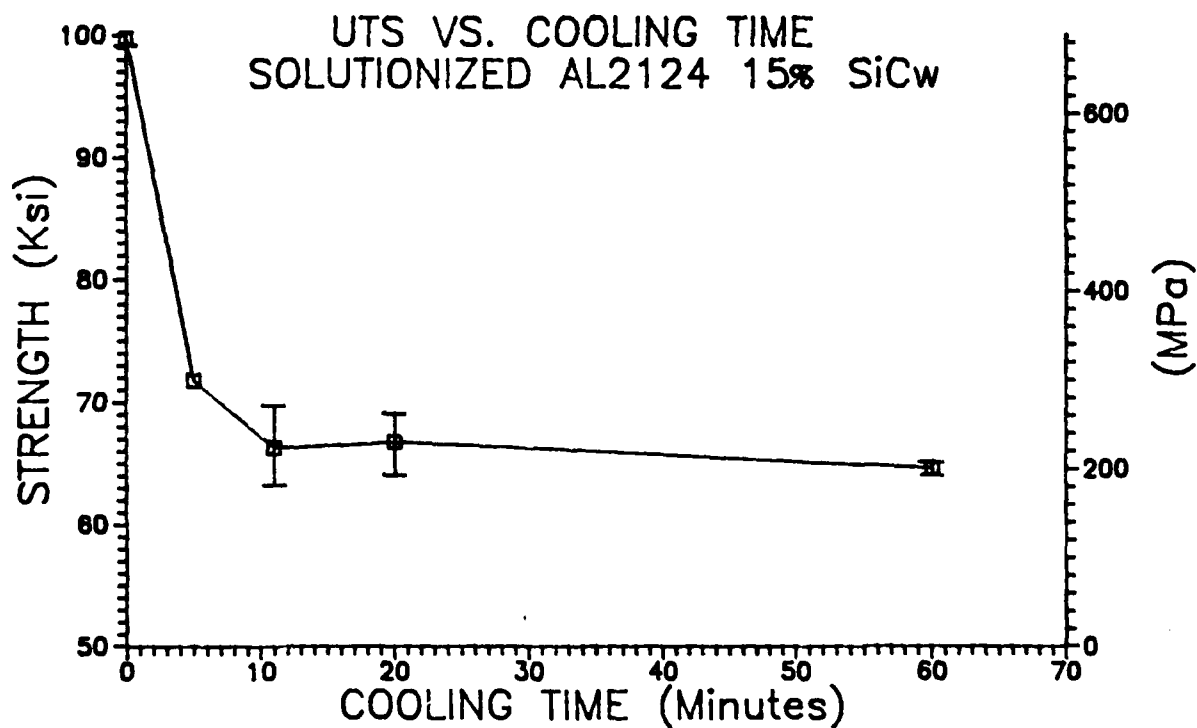
Fig. 8

ROLLED AL2124 15% SiCw
SOLUTIONIZED AND CWC

SAMPLES WERE SOLUTIONIZED
FOR 2 HOURS AT 500 DEGREES
CELSIUS BEFORE AGING

X-axis: ROOM TEMPERATURE AGEING TIME (hours)
Left Y-axis: ULTIMATE STRENGTH (Ksi)
Right Y-axis: (MPa)

Fig. 9

UTS VS. COOLING TIME
SOLUTIONIZED AL2124 15% SiCw

X-axis: COOLING TIME (Minutes)
Left Y-axis: STRENGTH (Ksi)
Right Y-axis: (MPa)

It was observed on the SEM that indeed the precipitates did grow back as the material cooled slowly. Figures 10, 11, 12 and 13 are compo photomicrographs of material that had been cooled for 5, 11, 22, and 60 minutes, respectively. One can see that the precipitates started out rather small and continued to grow larger. However, even the smaller precipitates caused the material to weaken substantially.

DETRIMENTAL EFFECTS OF LARGE PRECIPITATES

The next step is to show how the precipitates in the matrix actually weaken the composite. It is believed that there are three basic mechanisms in which weakening occurs. The growth of large precipitates tend to lower the shear strength of the matrix and they also tend to provide low energy crack paths or crack initiation sites. In addition, the precipitates tend to nucleate near whisker interfaces eventually covering the whisker and making it unable to carry much load.

LOWERING OF THE MATRIX SHEAR STRENGTH

When the precipitation hardenable matrix is grossly overaged, as in this case, only large precipitates exist and most of the copper is incorporated in these. Therefore, there is very little copper left to form the small, closely spaced precipitates needed to help prevent slip. Thus one is left with an almost pure aluminum matrix which is quite ductile and has a relatively low shear strength. However, when the composite material is solutionized and slightly aged, many small, closely spaced, precipitates form and dislocation motion, thus slip, is made more difficult. The matrix shear strength, therefore, would be greater in the T6 material.

Figure 10.   Compo   2000x
Cooled in 5 minutes.

Figure 11.   Compo   2000x
Cooled in 11 minutes.

Figure 12.   Compo   2000x
Cooled in 22 minutes.

Figure 13.   Compo   2000x
Cooled in 60 minutes.

Blanking shear tests were performed at room temperature on the material in order to compare the matrix shear strengths under the different conditions. These results could further be compared to blanking shear data for Al2024 and Al1100 which were given in the Mechanical Testing volume of the Metals Handbook (4). The room temperature results of the blanking shear tests are given in the following table.

BLANKING SHEAR DATA
Room Temperature

Material	Shear Strength (MPa)	(ksi)
Al2124 15% SiCw AR	181	26.3
Al2124 15% SiCw T6	319	46.3
Al2014 T6	284	41.2
Al1100 (commercially pure)	66	9.6

Note: These were the average of four punch tests
      of which the largest scatter was only 1.1 ksi.

It is noticed that the composite T6 material shows a higher shear strength than the T6 Al2014 alloy. This observation is probably due to the SiC whiskers themselves acting as barriers to dislocations which would tend to increase the shear strength. But most apparent is the dramatic effect the heat treatment has on the shear strength of the composite material. The shear strength of the T6 material is almost double that of the AR material.

The matrix shear strength is a very important factor in the strength of a composite, since it is the shear strength of the matrix that determines how well the matrix can transfer load to the reinforcement, assuming a strong

interface. The critical length to diameter ratio for a reinforcing fiber is

$$\frac{l_c}{d} = \frac{\sigma_{fmax}}{2\tau_{my}}$$

where σ_{fmax} is the maximum strength of the fiber and τ_{my} is the matrix shear strength (5). It is apparent that a higher matrix shear strength would mean that shorter fibers could carry more load, which would result in a stronger composite as is observed.

The difference in shear strengths of the AR and the T6 materials can also be seen by examining the fracture surfaces of samples that have been tensile tested in the longitudinal direction at room temperature. Figure 14 is the AR fracture surface which shows definite signs of low shear strength. The matrix appears to have been stretched and long whiskers have been pulled out of the surface. Figure 15, on the other hand, is the fracture surface of the T6 material, in which case evidence of high shear strength is shown. The microvoids are small with only short whisker ends inside them.

PRECIPITATES FORM CRACK INITIATION SITES AND PROVIDE
LOW ENERGY CRACK PATHS

The other way in which the precipitates weaken the material is by creating large voids in the material which can then act as crack initiation sites or provide a low energy crack path. The Al_2Cu precipitates are very brittle and weak and thus either the matrix plastically flows near stress concentration sites at the interface thus causing a large void at the interface or they simply fracture causing a large void between the halves. These interfacial voids and fractured precipitates can be seen very clearly in a
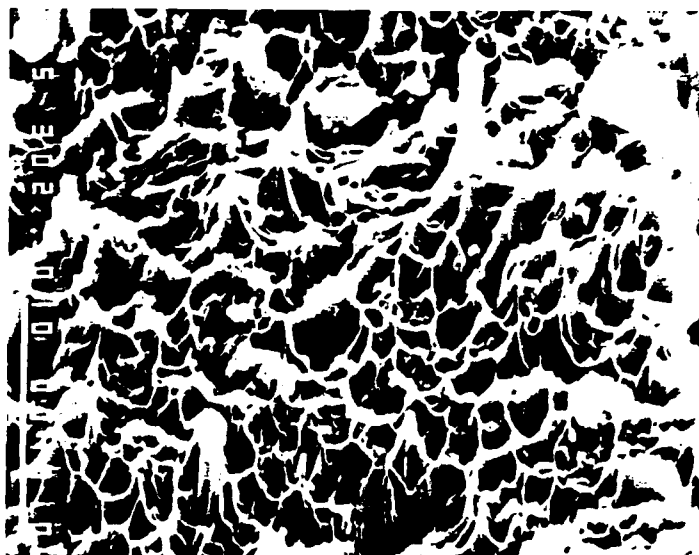
Figure 14.   SEM,   SEI,   3000x,   As Received, Room
             Temperature fracture surface: Shows
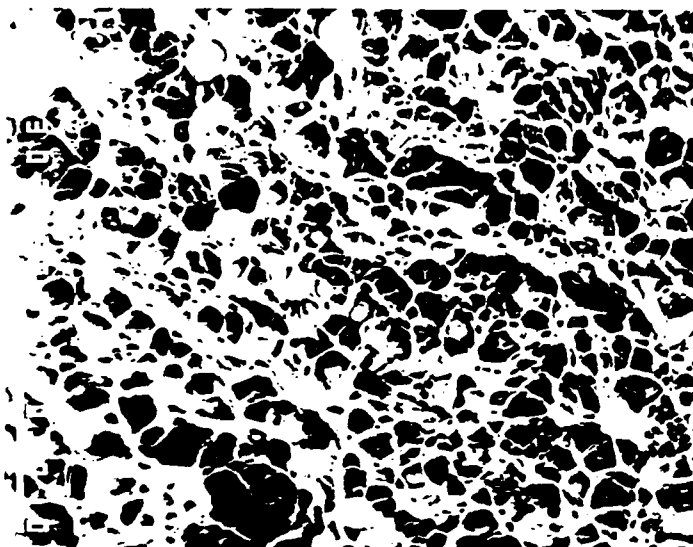   low shear and whisker pullout.

Figure 15.   SEM,   SEI,   3000x,   T6 , RT fracture
             surface: Microvoids are small with only
             whisker ends showing inside them.

compo mode photomicrograph of the cross section of AR material that had been tensile tested at room temperature (see Figure 16 and 17). Figure 18 shows a crack which is growing from a void in the AR material. Therefore, when stress is applied to this composite material large voids form near the precipitates and can form a crack. This crack can then easily grow by connecting the large voids resulting in fracture of the composite.

In order to observe the crack path, simple three point bend tests were performed. Unrounded tensile bars were cut to a length of 3 cm and two notches were cut 1 cm apart. This sample was set up as shown in Figure 19. The instron was used to slowly apply a force (0.005 cm/min) to the center of the sample. When a crack was observed to form at one of the notches, the instron was stopped and the sample removed. The cross section was then polished, ion milled, and studied on the SEM. The crack in the AR material can be seen to advance through the broken precipitates, as can be seen in Figures 20 and 21. It can be seen in Figure 22 that as the crack grows the stress field ahead of it tends to fracture more precipitates, thus further lengthening the crack.

In the T6 material, however, there are no large precipitates and thus the crack tip must instead advance through the much smaller voids at the ends of whiskers, as can be seen in Figures 23 and 24. This difference makes for a much stronger composite material.

The three point bend can also be carried out to fracture in order to study the fracture surfaces. These surfaces also show how the crack propagates. In the AR material many precipitates can be seen in the voids on the

Figure 16.
Compo 2000x:
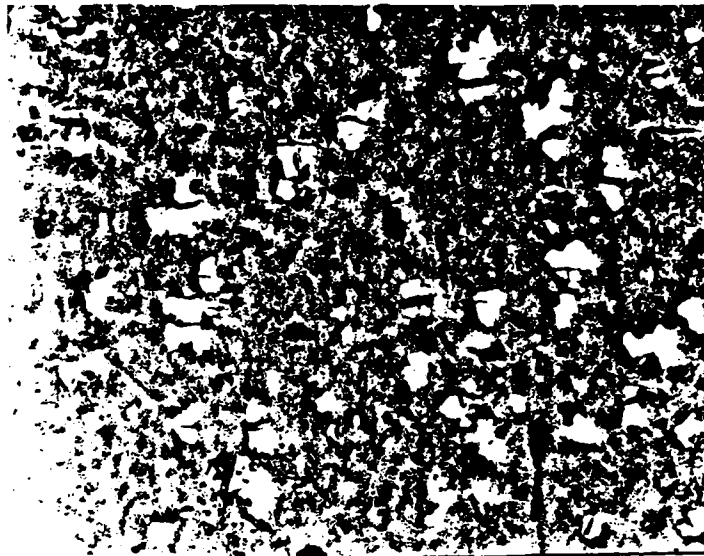Fractured precip.
and voids by their
side.

Figure 17.
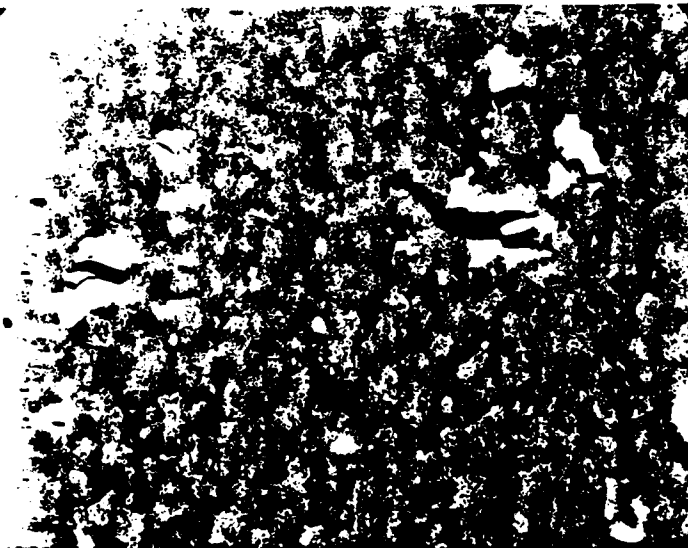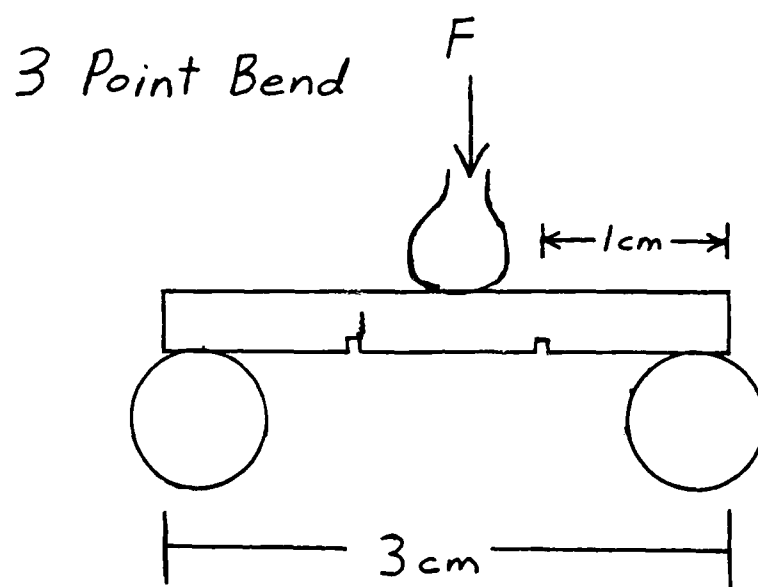Compo 1000x:
Many fractured
precipitates.

Figure 18.
Compo 2000x:
Crack growing from
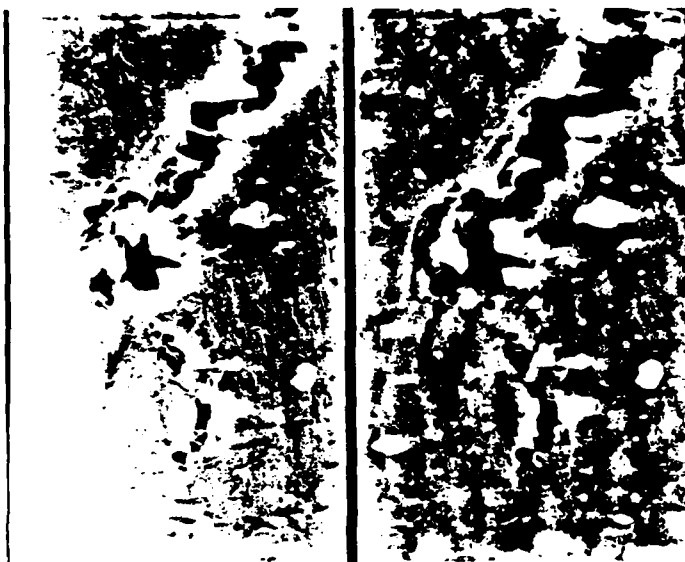fractured precip.

Figure 19.
3 POINT BEND SETUP

Figure 20.
SEI-Compo 1500x:
3 Pt. Bend crack
advancing from top
through broken
precipitates.

Figure 21.
Compo 2000x:
Crack being diverted
to the right through
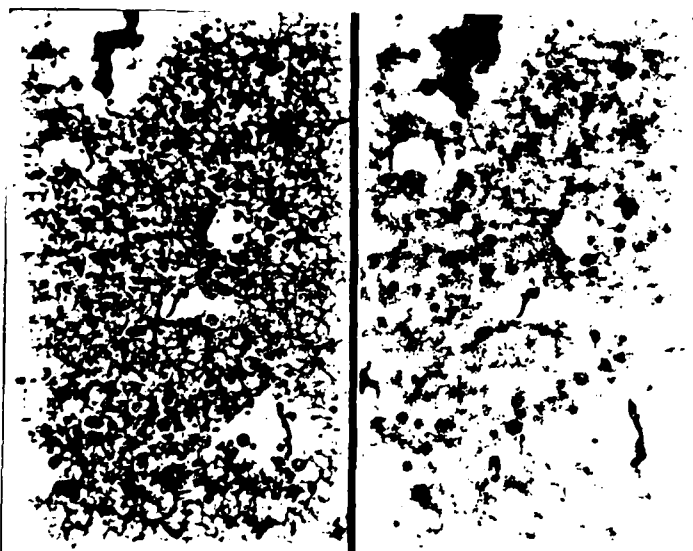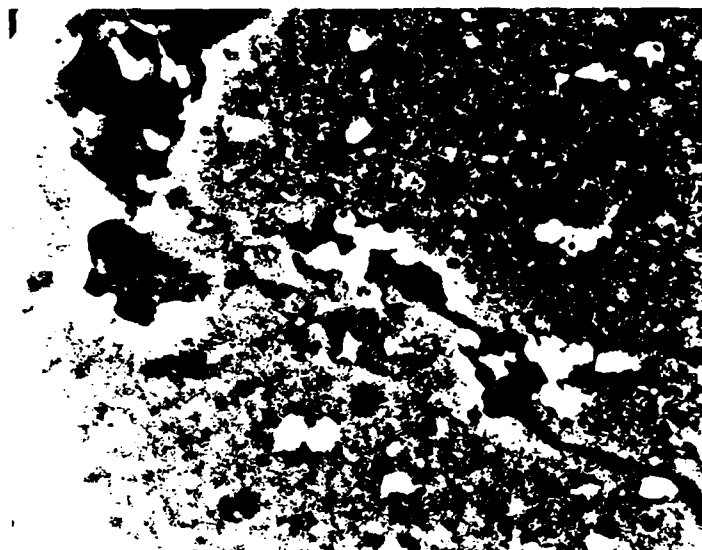broken precipitates.

Figure 22.
SEI-Compo 2000x:
Stress field ahead
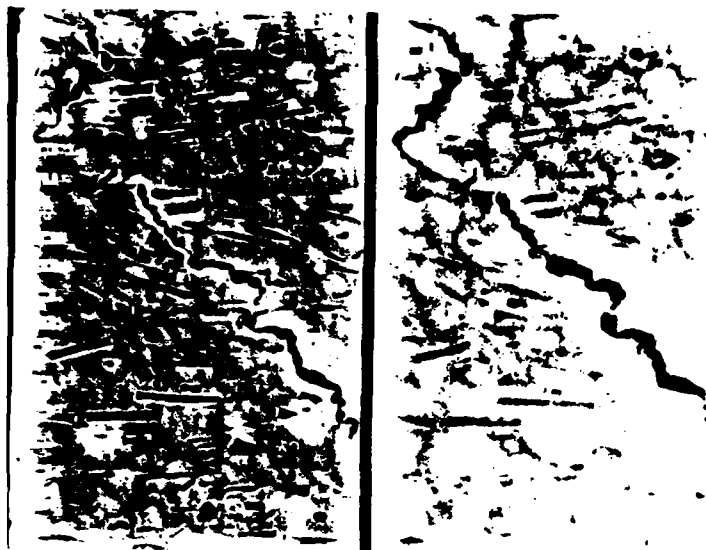of crack fractures
precipitates.

Figure 23.

SEI-Compo   2000x:

Crack advancing from top through voids at ends of whiskers.
Precipitates are dispersed, except for some very small ones.

Figure 24.

SEI   6000x:

Higher magnification of crack passing through whisker voids.

fracture surface as is shown in Figure 25. However, in the T6 material only whisker ends can be seen in the microvoids (see Figure 26).

NUCLEATION OF Al_2Cu NEAR THE WHISKER INTERFACE

The third way in which the precipitates weaken the composite is by nucleating at either the whisker interface or at dislocations near the interface. In order to observe the nucleation of the precipitates the cross sections of the samples that have been slowly cooled from the solutionizing temperature were studied. In these samples the precipitates were smaller and in their beginning stages of growth. Nearly all the precipitates are observed to be growing near a whisker interface, as can be seen in Figure 27. As the precipitate grows it begins to cover the interface of the whisker until the whisker is completely enveloped, as can be seen in Figure 28. The cross section of an AR sample observed parallel to the whiskers shows the advanced stages of precipitate growth very well (see Figure 29). One can see that the precipitates not only envelope one whisker, but they each tend to cover several whiskers.

When the whiskers are engulfed in brittle Al_2Cu they are unable to carry much load for two reasons. First of all, there appears to be no interfacial bonding between the whiskers and the Al_2Cu. Secondly, even if an interfacial bond did exist very little load could be transferred because the whisker and the Al_2Cu are both brittle materials. In fact many precipitates with whiskers pulled out of them were noticed on the fracture surfaces. Figure 30 shows a good example of how the whiskers simply pull out of the precipitates and are thus unable to help strengthen the material.
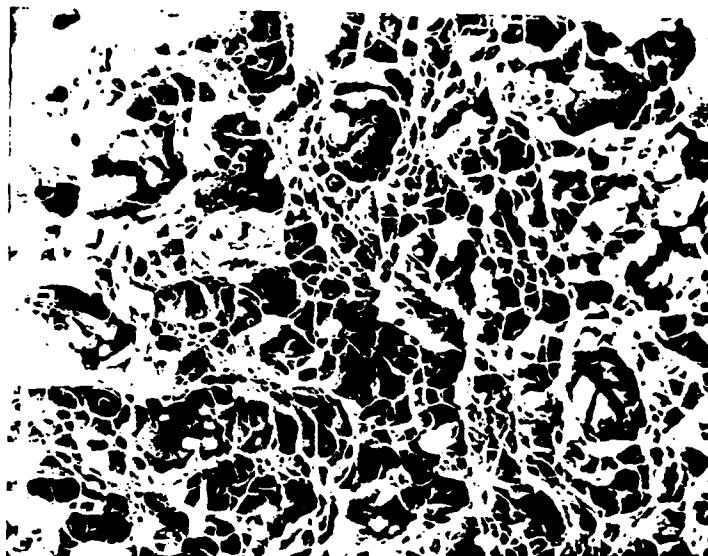
Figure 25.

SEI   2000x   AR material:

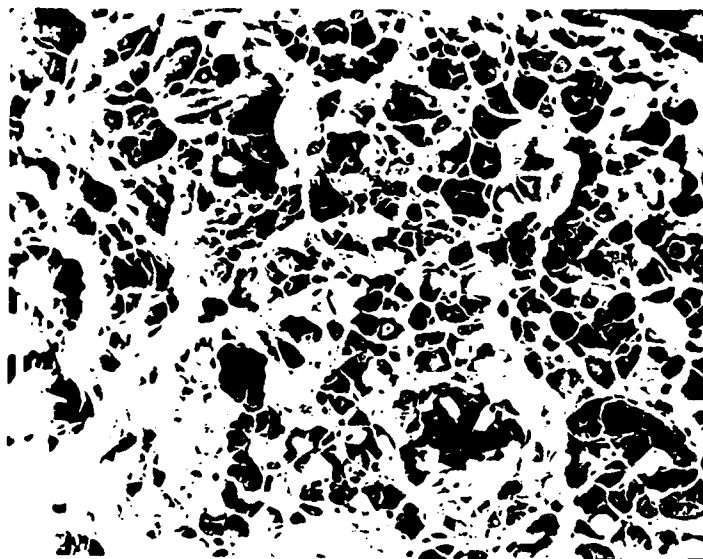3 Pt. Bend fracture surface with many precipitates exposed.

Figure 26.

SEI   3000x   T6 material:

3 Pt. Bend fracture surface showing small voids with whisker
ends in them. No precipitates can be seen.

One question left unanswered is, do the precipitates nucleate at the interface or at dislocations near the interface? The answer can be found by studying the extruded Al2124 20% SiCw composite on the SEM. Figure 31 and 32 are compo photomicrographs of this material. It is apparent that the precipitates seem to grow in linear groups along the extruded direction. From the photomicrographs one can see that the whiskers are distributed rather evenly throughout the material even though the precipitates are not. This would tend to imply that the the precipitates are growing at dislocations and that some areas of the extruded material have higher dislocation densities than others. Some areas probably have higher dislocation densities due to an uneven distribution of stresses which occurs during the extrusion process. Thus, plastic deformation would be uneven in the extruded composite.

GROWTH OF PRECIPITATES

The rate of precipitate growth should reach a maximum at some temperature. In order to find this temperature DSC (Differential Scanning Calorimeter) experiments were performed. Figure 33 shows the results of DSC analysis performed on material that had been solutionized. It is noticed that the precipitates seem to be growing fastest at a temperature of about 250°C for the following reason: According to the phase diagram for copper in aluminum, at 4 wt.% Cu the aluminum is supersaturated with copper up to a temperature of about 500°C, see Figure 34 (6). Therefore, it is energetically favorable for theta phase precipitates to form at temperatures below 500°C, and thus energy is released upon their formation (exotherm). This energy then goes to heat the material. Therefore, when the precipitates

Figure 30.
SEI 10000x:
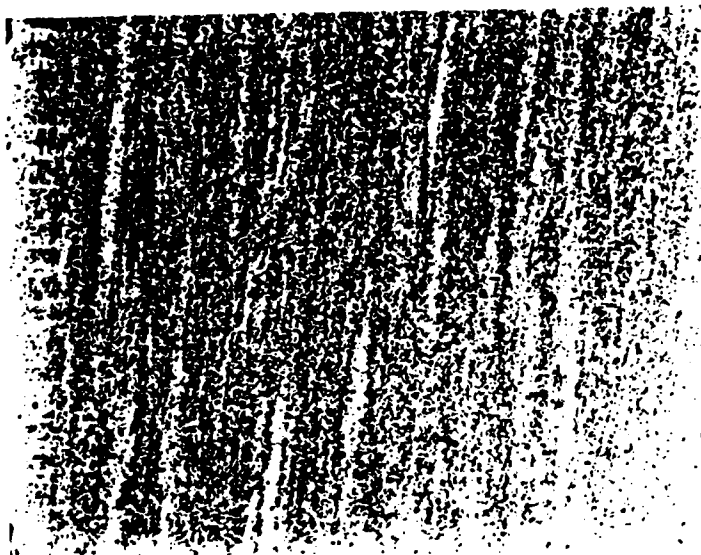Large precipitate
with whiskers
pulled out of it.

Figure 31.
Compo 120x
Extruded material:
Precipitates are
growing in linear
bunches along ext
direction.

Figure 32.
Compo 480x:
Whiskers are distributed
rather evenly in the
extruded material even
though the precipitates
are not.

Figure 33.   Differential Scanning Calorimetry (DSC) results
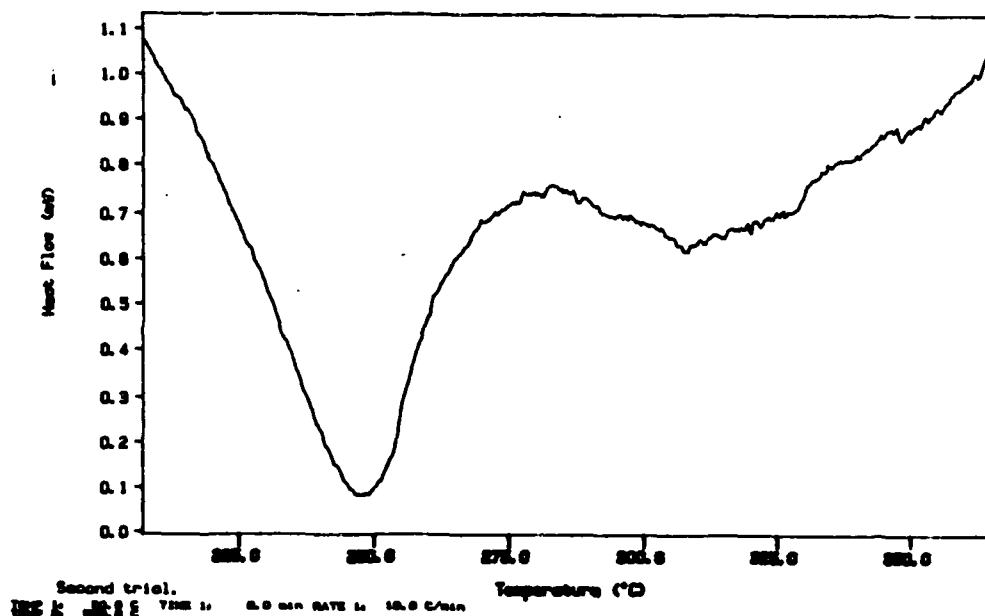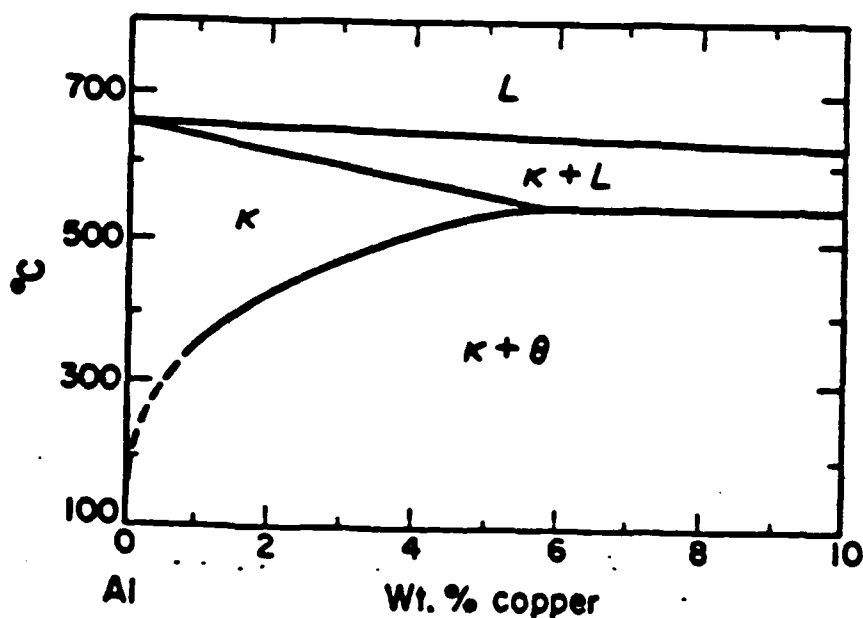for solutionized rolled Al2124 15% SiCw.

Figure 34.   Phase diagram for the aluminum-copper system.
The pure θ phase contains about 46 wt.% Cu at
room temperature and corresponds to $CuAl_2$ (6).

are growing fastest it should require a minimum amount of heat per gram to raise the temperature at a fixed rate. From the DSC graphs it can be seen that there is a minimum heat per gram at a temperature of 250°C, which implies the precipitates are nucleating and growing fastest at this temperature.

EFFECTS OF HIGH TEMPERATURE ON Al 2124-SiCw COMPOSITES

Figure 35, displays the general trend of aluminum composites at elevated temperatures. It is apparent that the strength of these composites drops off sharply at temperatures over 300°C. This observation raised the question as to what is the controlling mechanism for failure at these higher temperatures and if the aluminum 2124/SiCw composites displayed similar behavior. Hence, the second goal of this research was to define the mechanical response and fracture characteristics of Al2124/SiCw composites at elevated temperatures and how they are related to the microstructure.

EFFECTS OF TEMPERATURE ON THE STRENGTH

Figure 36 shows the results of the Al2124/SiCw tensile tests. Before these tests were performed the sample was allowed to heat up to the desired temperature (about 15 minutes) and then were held at that temperature for at least 15 minutes to ensure equilibrium. Also included in this graph is data for extruded Al 1100/20 v/o SiCw. It can be seen that the Al2124-SiC composite seems to hold its strength to slightly higher temperatures than the composites in Figure 35. The strength did not drop sharply until a temperature of 300°C was reached. It is also noticed that the precipitation hardened alloy composite had a much higher

UTS vs. Temperature
For Various Composites

7075 30% SiCw
6061 30% SiCw
356 20% SiCr
6061 20% Al2O3
332 18% Al2O3
1100 20% SiCw

(MPa)

TEMPERATURE (degrees C)

STRENGTH (ksi)

Figure 35.
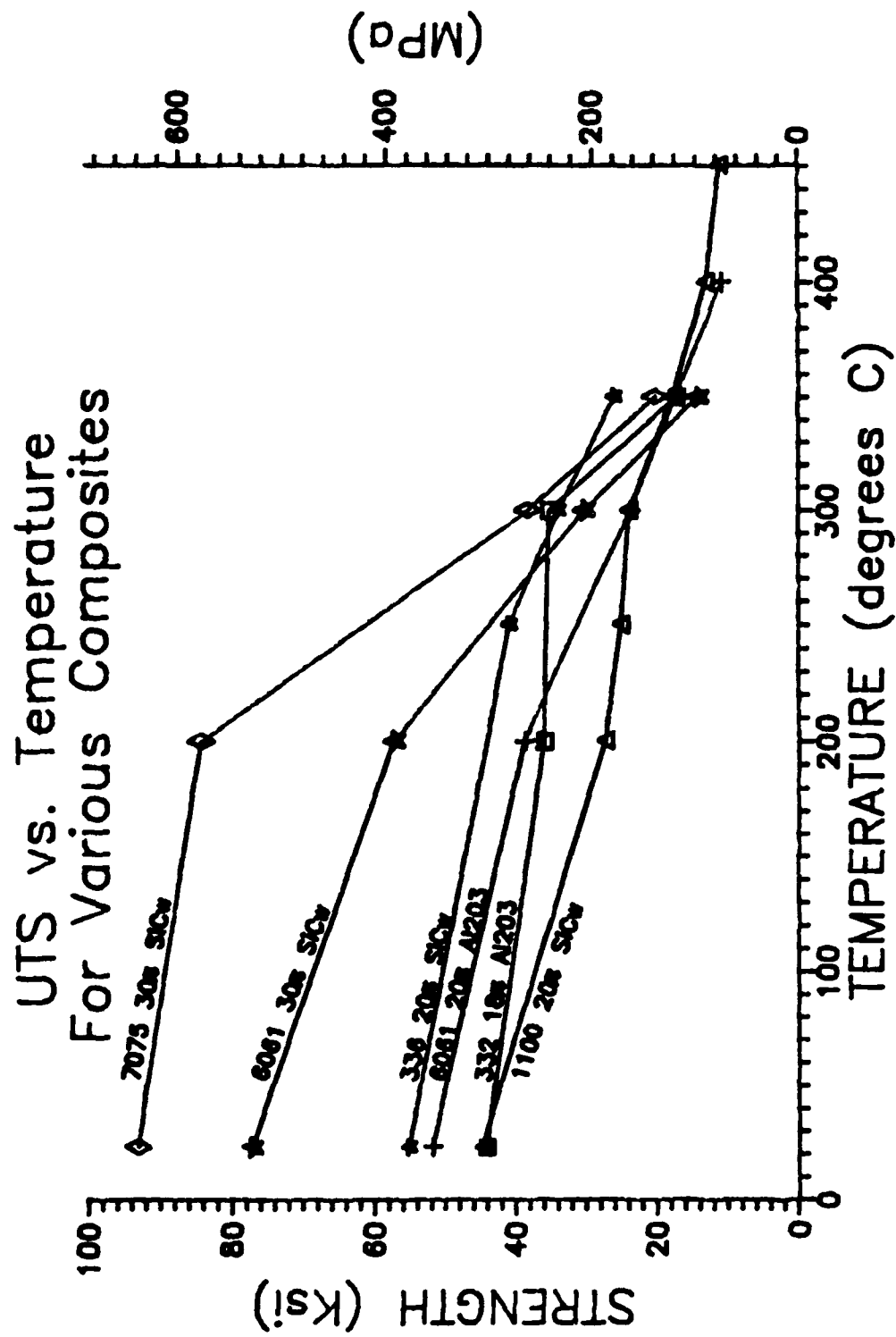
Fig. 36

UTS vs. Temperature
Rolled Al 2124 15% SiCw

T6 Longitudinal

T6 Transverse

AR Longitudinal

AR Transverse

Ext Al 1100 20% SiCw

STRENGTH (ksi)

(MPa)

TEMPERATURE (degrees C)
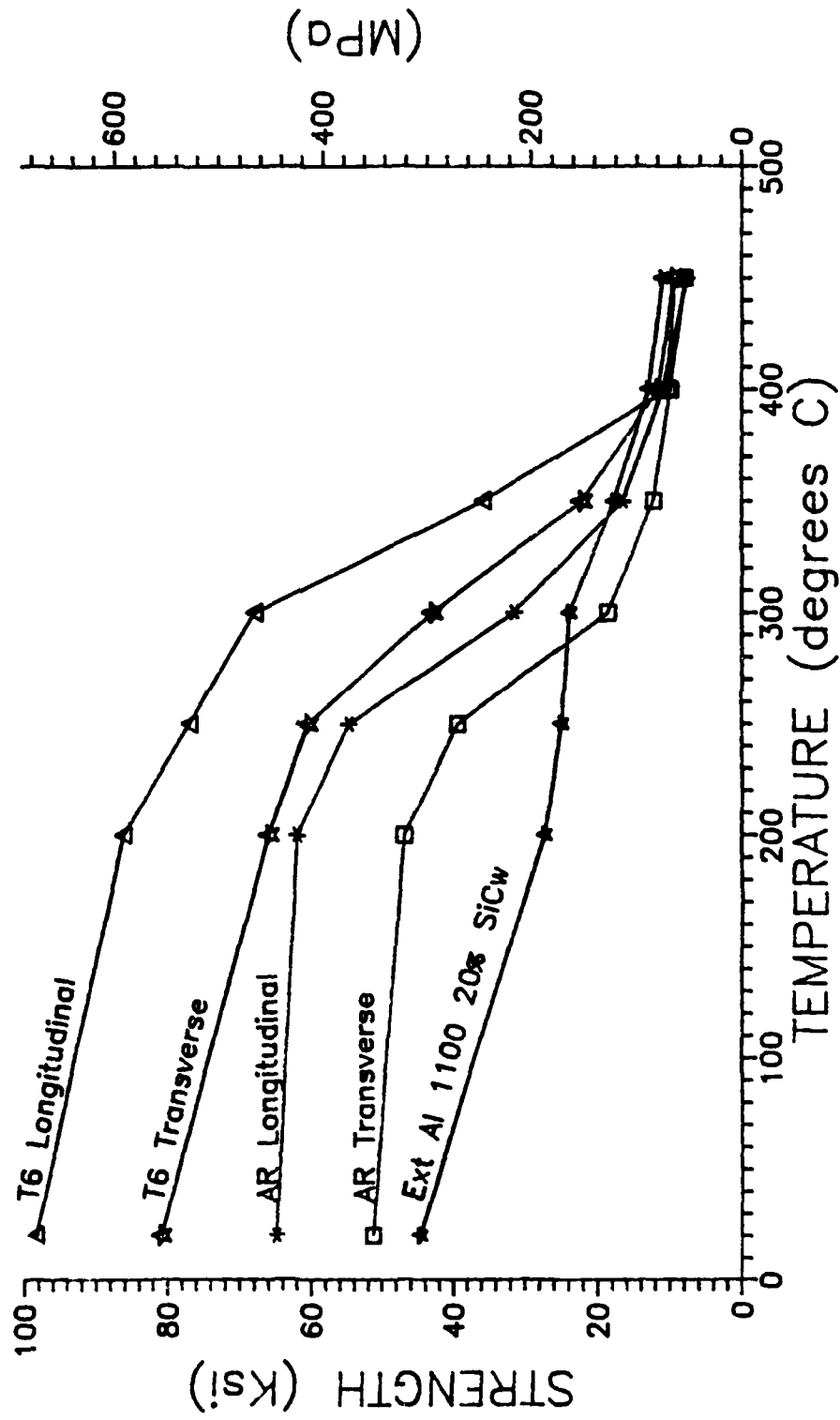
strength than the commercially pure aluminum matrix composite at temperatures up to $350^{O}C$. At temperatures over $400^{O}C$ it appears as if the precipitates offer no contribution to the strength of the composite, as one would expect since the precipitates are solutionized and offer no barrier to slip. In fact the orientation of the reinforcement itself does not seem to be a factor in the strength at temperatures above $350^{O}C$. Thus implying that the reinforcement does not carry any load and does not, therefore, contribute to the strength at such high temperatures.

Figure 37 shows data for extruded Al2124/20 v/o SiCw that has undergone a T6 heat treatment and for unreinforced extruded Al2124 also with a T6 heat treatment. In this case, the SiC whisker reinforcement gives a much stronger material up to a temperature of $300^{O}C$. However, above this temperature the strengths are nearly identical, again implying that the whiskers basically carry no load at temperatures at and above $300^{O}C$. It is also noticed that the hot rolled material seems to maintain its strength a little better at higher temperatures, possibly due to the higher dislocation density of the rolled material due to work hardening. The dislocations themselves would help prevent slip and give the matrix a greater shear strength at higher temperatures.

It is rather obvious that the strengths of the aluminum composites drop off at higher temperatures simply due to a loss in shear strength of the matrix. It can be seen from a graph of shear strength vs. temperature (Figure 38) that the shear strength drops off rather quickly with an increase in temperature. At temperatures over $200^{O}C$ the shear strength of the T6 and the AR material both fall to nearly identical
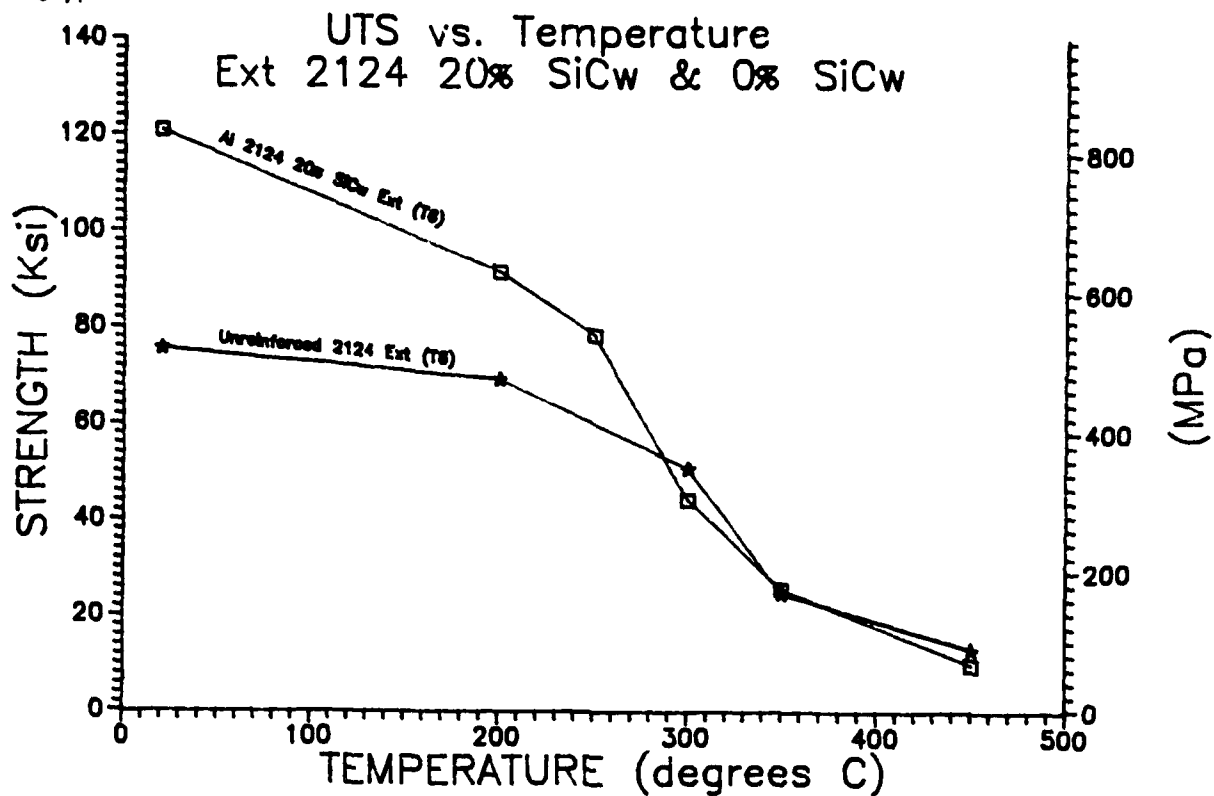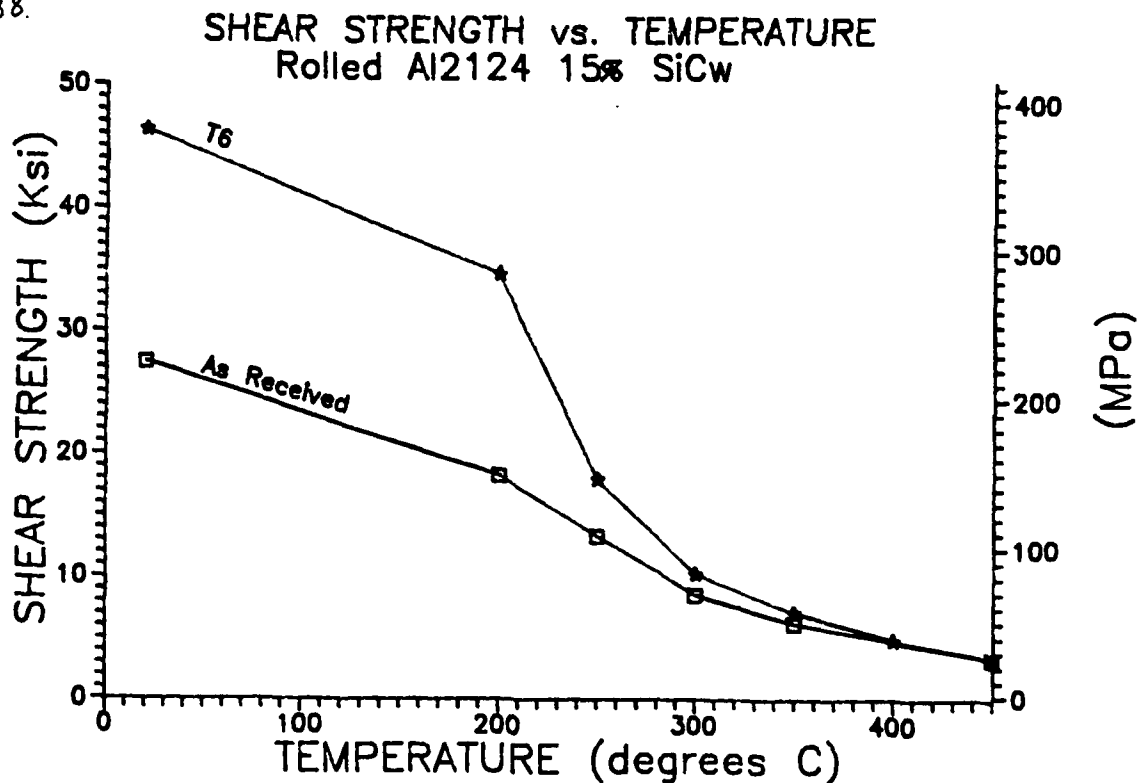
Figure 37.

UTS vs. Temperature
Ext 2124 20% SiCw & 0% SiCw

Al 2124 20% SiCw Ext (T6)

Unreinforced 2124 Ext (T6)

Figure 38.

SHEAR STRENGTH vs. TEMPERATURE
Rolled Al2124 15% SiCw

T6

As Received

values. Recall that the UTS of the T6 transverse material did not drop off sharply until a temperature of $250^{\circ}C$ was reached. Therefore, the shear strength should not be expected to drop off in the T6 material until a temperature of $250^{\circ}C$ is also reached. Thus, there appears to be a discrepancy between the behavior of the UTS and the shear data. However, one must keep in mind that the shear samples were heated much more slowly and were held at the desired temperature longer than the tensile tested samples. Thus, the precipitates were allowed to form for a longer time which would result in the lower temperature drop of the shear strength that is observed.

Lowering of the shear strength at higher temperatures can also be distinguished by studying whisker void sizes at the different temperatures. Figures 39, 40 and 41 are photomicrographs of cross sections of rolled 2124 near the fracture surface of tensile tested specimens. At room temperature the voids at the whisker ends are very small and in their beginning stages. In fact they are starting at the stress concentration sites at the edges of the whiskers as is shown in a paper by Nutt (7). However when the temperature is raised to $450^{\circ}C$ the voids become much larger and are at times as long as the length of the whisker itself. The shear strength of the matrix is so low that the whiskers are literally pulled through the matrix and contribute nearly nothing to the strength.

EFFECTS OF TEMPERATURE ON DUCTILITY

Some rather interesting phenomena happen to the ductility of the rolled aluminum matrix composite at elevated temperatures. Figure 42 and 43 are graphs of strain vs. temperature for the rolled material tensile

Figure 39.
SEI 2600x
T6 material
tensile tested
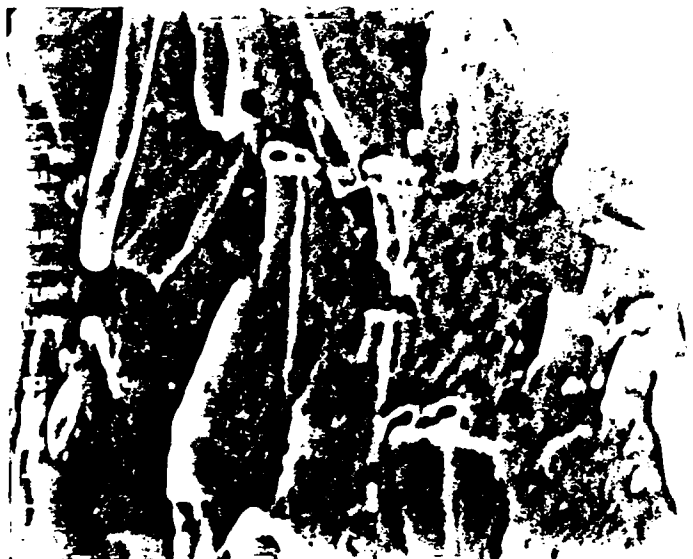at room temp.
Voids are small.

Figure 40.
SEI 10000x:
T6 material
tensile tested
at RT. Voids are
starting at the
edges of whiskers

Figure 41.
SEI 2000x:
T6 material
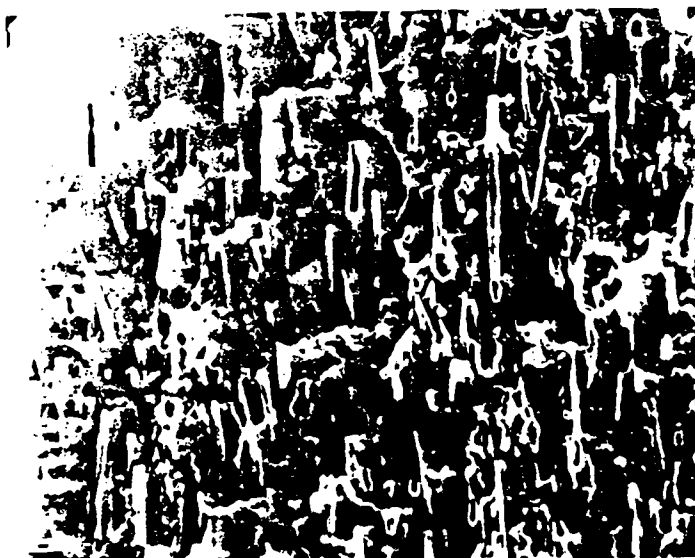tested at 450°C.
Voids are very long.

Figure 42.

STRAIN vs. TEMPERATURE
Rolled Al2124 Longitudinal

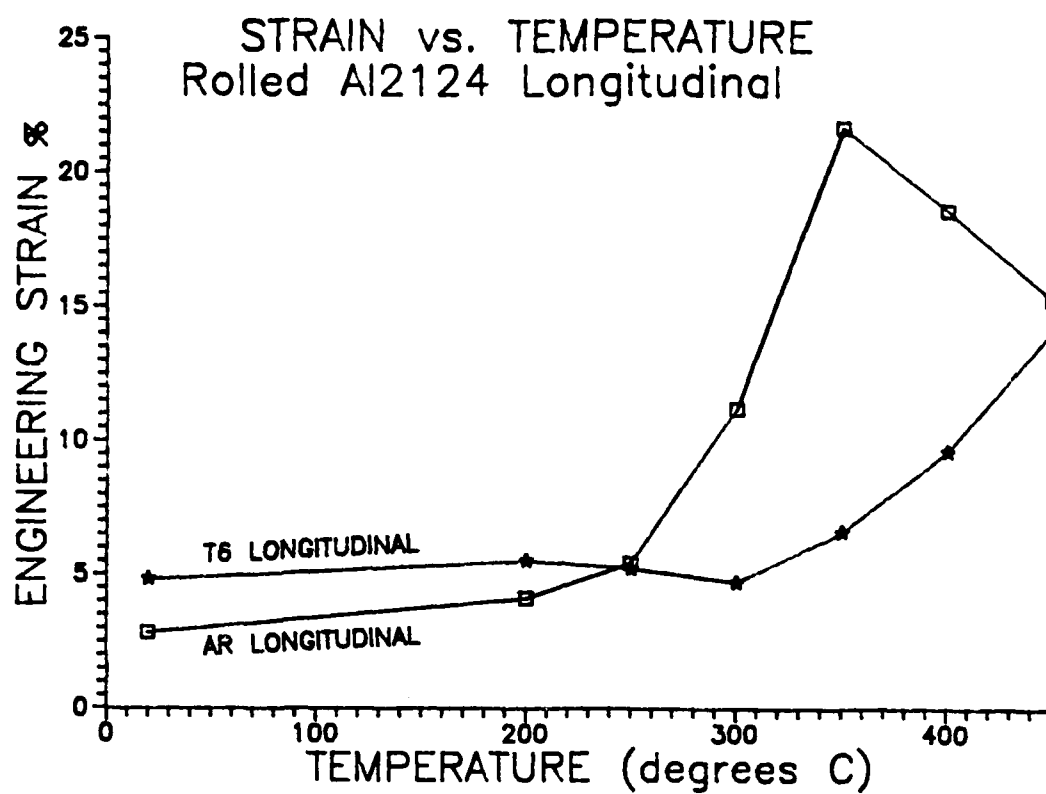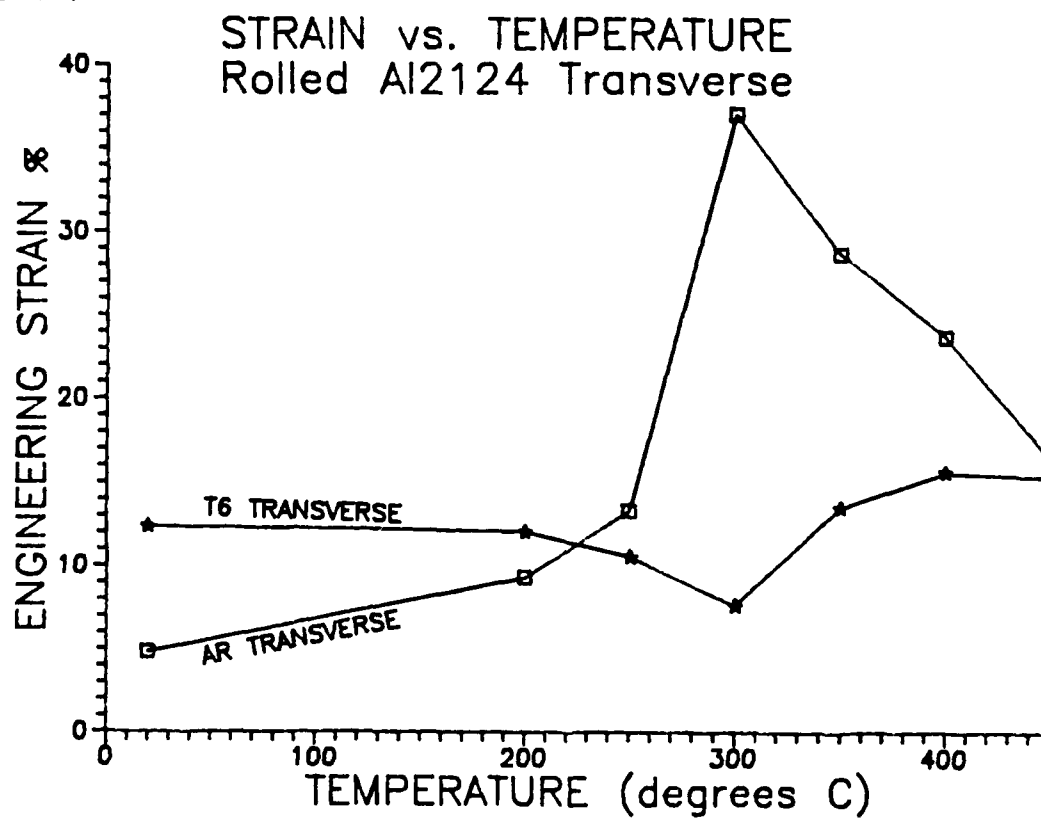Figure 43.

STRAIN vs. TEMPERATURE
Rolled Al2124 Transverse

tested in the longitudinal and transverse directions. The first thing to notice is that the strain in the transverse direction is generally much higher than that in the longitudinal direction, as one would expect since the whiskers constrain the material much more in the longitudinal direction. However, at a temperature of 450°C the whiskers no longer constrain the matrix thus the strain is the same for both the AR and the T6 material in both the transverse and longitudinal directions.

At room temperature it is believed that the AR material is less ductile simply because the large precipitates act as crack initiation sites, thus very little plastic deformation is allowed to occur before fracture. The lack of plastic deformation can readily be seen either by observing the room temperature stress strain curves for the AR and the T6 material in Figures 44 and 45, or by looking at the Yield Strength vs. Temperature graph in Figure 46. One can see that the room temperature yield strengths are the same even though the ultimate strength of the T6 material is 55% higher than that of the AR material. Thus very little plastic strain takes place in the AR material before fracture.

In the T6 material, as the temperature is increased the material becomes less ductile and reaches a minimum strain at 300°C. It is believed that this minimum is due to a nucleation and growth of larger precipitates which would again act as crack initiators and decrease the plastic deformation, thus decreasing the strain. This decrease in plastic deformation is made obvious by comparing the number of voids created at the ends of whiskers in the material tested at the different temperatures. Figures 47, 48 and 49 are photomicrographs of general areas in samples tested at

Figure 45.

Room temperature stress-strain
curve for T6 material: Much
more plastic flow.

Figure 44.

Room temperature stress-strain
curve for AR material: Very
little plastic flow.

Figure 46.

0.2% YIELD STRENGTH vs. TEMPERATURE
Rolled AL2124 15% SiCw
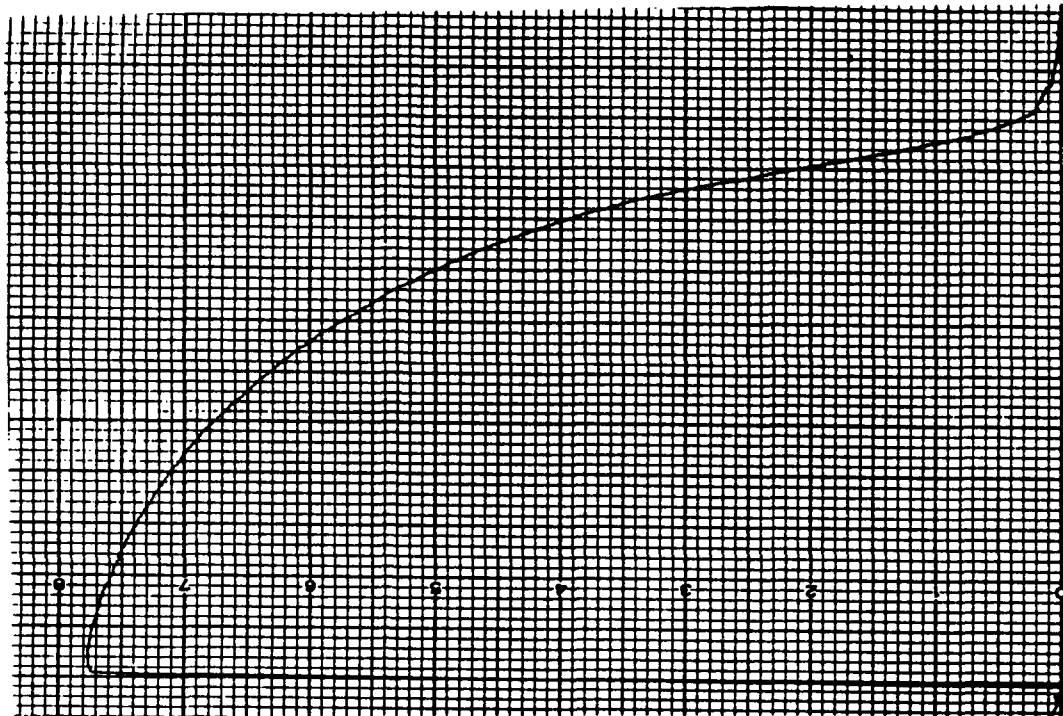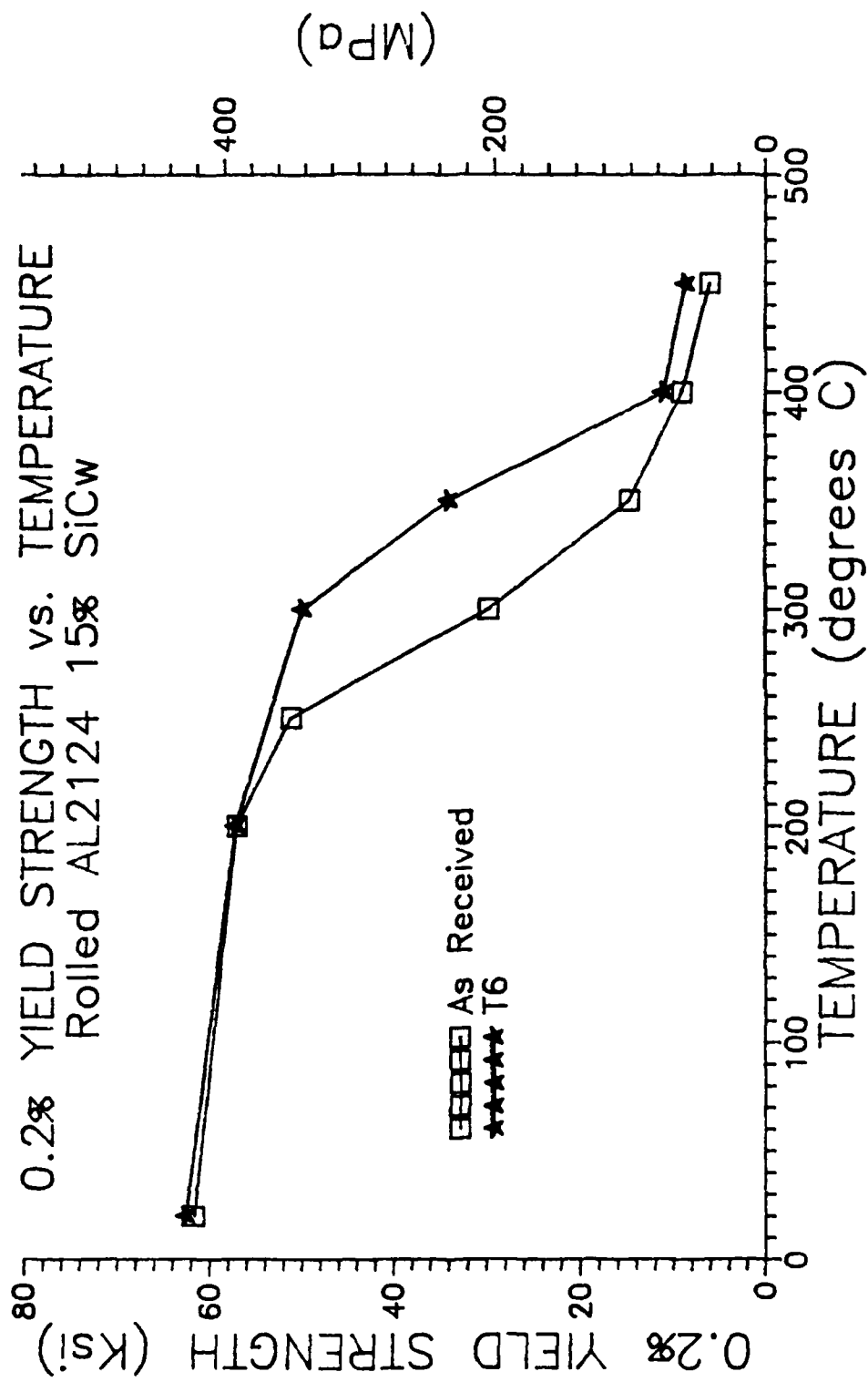
Figure 47.
SEI   1000x:
T6 material
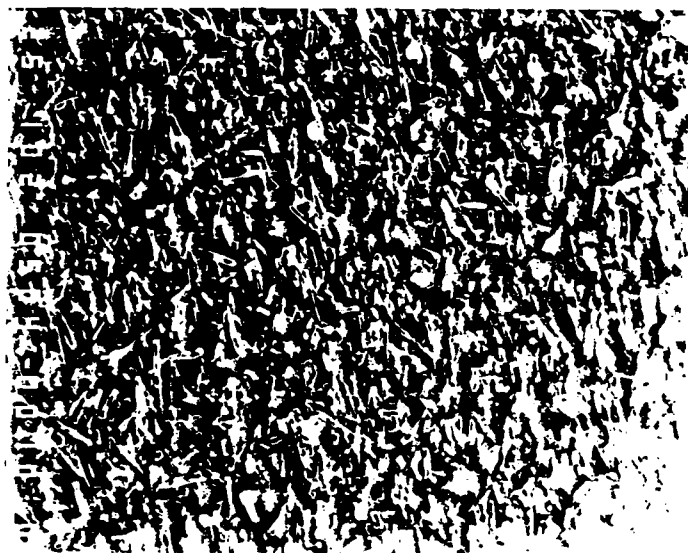tested at 250°C.
Very many voids.

Figure 48.
SEI   1000x:
T6 material
tested at 300°C.
Few voids.

Figure 49.
SEI   1000x:
T6 material
tested at 350°C.
Many voids again.

250, 300, and 350 degrees celsius, respectively. It is clear that there are very few whisker voids in the material tested at 300°C compared to the others. A count gave 98 and 127 voids in the material tested at 250°C and 350°C, respectively. However the material tested at 300°C only had 33 voids. Thus, plastic flow is at a minimum at 300°C in the T6 material.

The strain of the AR material, on the other hand, increases to a maximum at about 350°C. This increase in strain is likely due to the large existing precipitates growing even larger and the matrix thus becoming even more ductile combined with the matrix becoming more ductile with the increase in temperature alone. This increase in ductility also corresponds to a minimum in yield strength, as can be seen in Figure 46. The large precipitates do not initiate cracks as readily when the matrix is more ductile, simply because the yield strength is much less, which in turn, increases the fracture toughness. The strain decreases again as the temperature is raised to 450°C possibly because this high temperature solutionizes the precipitates thus resulting in some solid solution strengthening which makes the matrix less ductile.

The degree of necking of the tensile specimens also represents the ductility of the composite. Figures 50 and 51 are the graphs of necking vs temperature for transverse and longitudinal tensile tests. As one would expect after studying the strain data, the necking was generally less in the longitudinal samples. Also the T6 samples tend to resist the necking to higher temperatures.

Thus the precipitation hardenable matrix not only has a large effect on the strength of the composite but it also has a large effect on the ductility. Not only does heat

Figure 50.

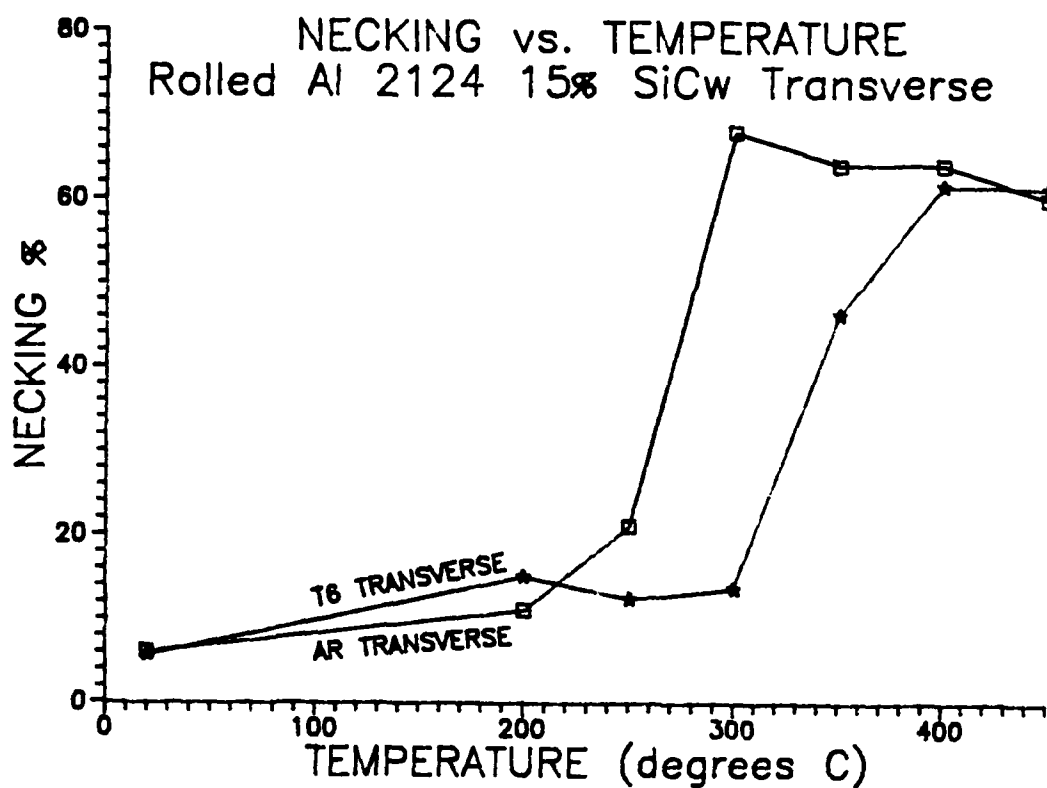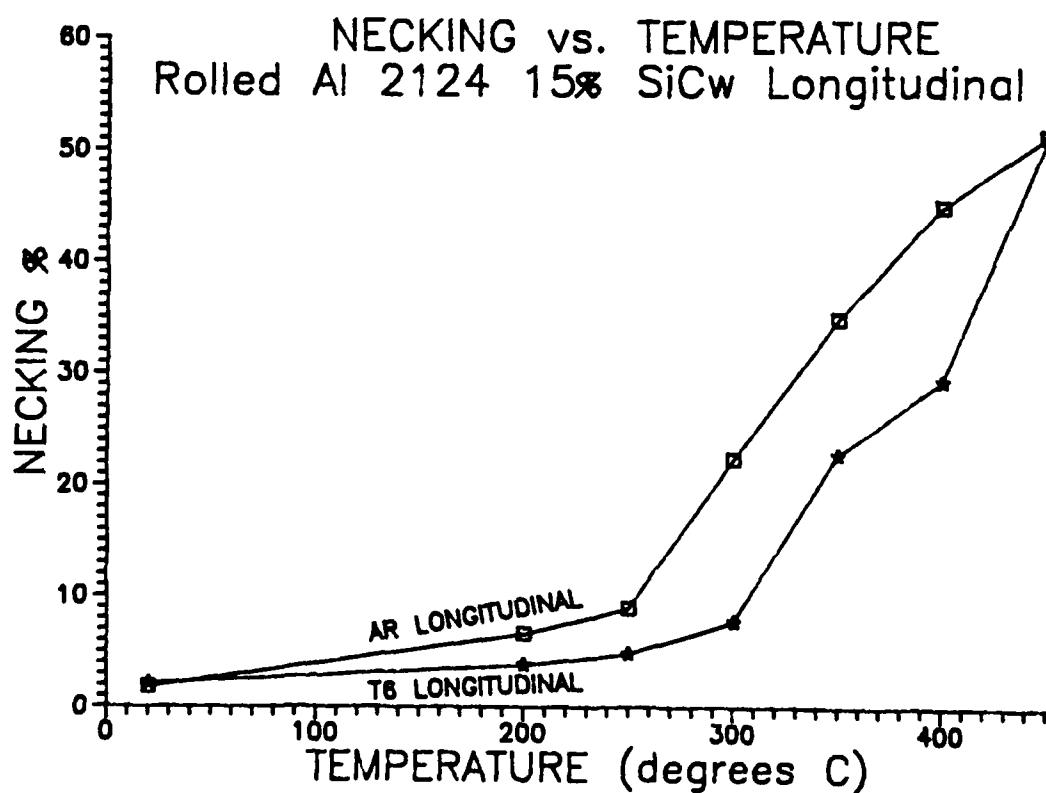**NECKING vs. TEMPERATURE**
Rolled Al 2124 15% SiCw Transverse

Figure 51.

NECKING vs. TEMPERATURE
Rolled Al 2124 15% SiCw Longitudinal

treating the rolled material strengthen it, but it also increases its ductility at room temperature.

SECTION III
CONCLUSIONS

EFFECTS OF HOT ROLLING ON AL2124-SiCw COMPOSITES

It has been confirmed in this study that when Al2124 15% SiCw is hot rolled it loses a good portion of its strength. It has been found that this loss in strength is mainly due to overaging of the precipitation hardenable matrix. Evidence shows that large Al_2Cu precipitates grow as the material is allowed to cool down after the rolling step.

It has also been confirmed that the strength of the material can be regained by subjecting it to a T6 heat treatment (1 hour at $500^{\circ}C$, CWC, and aged at $150^{\circ}C$ for 8 hours). Experiments show that the strength is regained mainly because the heat treatment disperses the precipitates. However, it has been discovered that this rather long heat treatment sequence can be made much shorter due to the high dislocation density of this material. The precipitates can be dispersed by solutionizing the material at $500^{\circ}C$ for very short periods of time (as short as 1.5 minutes) followed by cold water quenching. Likewise, it has been found that only a very simple aging process is needed to obtain peak strength. Artificial aging at $150^{\circ}C$ is not necessary. The material need only be aged at room temperature for a time of 7 hours or more. Thus industry should be able to heat treat the rolled composite material much more efficiently and inexpensively than a T6 heat treatment would permit.

WAYS IN WHICH THE PRECIPITATES WEAKEN THE COMPOSITE

It was discovered that there are three basic ways in

which the large precipitates weaken the composite.

1) The growth of large precipitates can weaken the material by removing the small precipitates which are needed to help prevent dislocation motion, thus slip. Therefore, the shear strength of the matrix is reduced which results in a larger critical whisker length. Thus shorter whiskers are unable to carry as much load and the material is weakened.

2) The large precipitates also tend to create large voids in the composite which then act as crack initiation sites and also provide low energy crack paths.

3) Lastly, evidence shows that the precipitates tend to nucleate at dislocations near the interface. Therefore, the precipitates tend to grow around the whiskers and envelope them, thus rendering the strengthening effect of the whiskers useless.

HIGH TEMPERATURE FRACTURE

It was shown that the Al2124 matrix provided a much stronger composite than a pure aluminum matrix, at least up to a temperature of $400^{\circ}C$. At temperatures higher than this it appears as if precipitation strengthening offers no contribution to the strength. It has also been shown that the reinforcement itself offers no contribution to the strength at temperatures above $300^{\circ}C$, due to very low shear strengths in the matrix at these high temperatures. Through elevated temperature shear tests it was shown that the main reason for the drop in strength at higher temperatures was due to a rapid decrease in shear strength of the matrix.

HIGH TEMPERATURE DUCTILITY

It was found that the strain in the transverse direction was much larger than that in the longitudinal direction. It was also found that not only does the T6 heat

treatment increase the strength of the rolled composite but it also increases the room temperature ductility. The AR material has very poor ductility because the large precipitates initiate cracks, which lead to tensile fracture, before much plastic deformation can take place. However, the ductility of the AR material increases with temperature to a maximum near 350°C, while the ductility of the T6 material decreases to a minimum at a temperature of 300°C. The precipitates are also thought to be responsible for these effects.

OTHER OBSERVATIONS

It was also observed using Differential Scanning Calorimetry that the precipitates seem to nucleate and grow fastest at a temperature of 250°C.

The extruded Al2124 15% SiCw composite was much less homogeneous than the hot rolled material. Precipitates were observed to grow in linear groups along the extruded direction apparently due to variations in plastic deformation.

The rolled T6 material resisted necking to higher temperatures than the AR material. The AR material began to neck significantly at 250°C while the T6 material resisted the necking until a temperature of 300°C was reached.

SECTION IV
FUTURE RESEARCH

From this research, it is obvious that the matrix plays a crucial role in the properties of a composite, both at high and low temperatures. The shear strength in particular is very important since it is the main factor in how much load the whiskers will carry. One can see that a precipitation hardenable matrix such as Al 2124 does make a very good matrix at temperatures below 200°C, however, it does not make a very good matrix at high temperatures since the copper precipitates out and leaves behind almost pure aluminum which has a low shear strength.

It is believed that in order to make a strong high temperature aluminum matrix composite one should use dispersoid hardening rather than precipitation hardening. The dispersoids should be very small (.01 - .1 μm) and insoluble at high temperatures. They should also have an incoherent interface so that dislocations will be forced to bypass them. A suggestion for these dispersoids might be ground up SiC particles, since these would satisfy the above requirements. These dispersoids would then work harden the matrix even at high temperatures (although not by as much since dislocations can bypass particles faster at high temperatures). The dispersoids would probably strengthen the composite at higher temperatures, however, the problems of low ductility and fracture toughness would probably still exist.

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PUBLICATIONS AND PRESENTATIONS

A. PUBLICATIONS

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Accepted for Publication

F. E. Wawner and J. W. McCoy, "The Origin of the 'Crack Tip' Mode of Failure in Boron Filaments," Accepted for publication in Journal of Materials Science (1988).

Submitted for Publication

F. E. Wawner, "Discontinuously Reinforced Metal Matrix Composites: Microstructure and Mechanical Property Correlations," Invited Contribution to the *Encyclopedia of Composites*, Edited by S. M. Lee, VCH Publishers, New York, 1988.

To Be Submitted

R. Schueller and F. Wawner, "An Analysis of High Temperature Behavior of Discontinuously Reinforced Al Alloys," To Scripta Met.

R. Schueller and F. Wawner, "An Analysis of the Effects of Hot Rolling on Discontinuously Reinforced Al Alloys," To Met. Trans. A.

B. PRESENTATIONS

R. Schueller and F. Wawner, "An Analysis of High Temperature Behavior of Discontinuously Reinforced Al Alloys," Presented at the 10th Annual Dicontinuous Reinforced Metals Working Group Meeting, Park City, UT, Jan. 4-8, 1988; Published in Preceedings.

R. Schueller and F. Wawner, "An Analysis of the Effects of Hot Rolling on Discontinuously Reinforced Al Alloys," Presented at the 66th Annual Meeting of the Virginia Academy of Science (University of Virginia, Charlottesville, VA, May 25-27, 1988).

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